# **SILICON-GLASS ANODIC BONDING**

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Key words: anodic bonding, silicon, Pyrex glass, piezoresistive pressure sensor

Abstract: Piezoresistive pressure sensors test structures fabricated on the same silicon wafer were anodic bonded to Pyrex glass wafer and bonding characteristics were analyzed. Surface profiles of both, silicon and Pyrex wafer, were measured before and after bonding process using Taylor-Hobson Talysurf surface profiler. A simple test method for non-destructive in-situ evaluation of both, anodic bonding parameters and bond quality was introduced. Possible causes forthin silicon diaphragms deflection, detected after anodic bonding process, were analyzed and discussed.

# **Anodno bondiranje silicij - steklo**

Kjučne besede: anodno bondiranje, silicij, Pyrex steklo, piezorezistivni senzor tlaka

Izvleček: Testne strukture piezorezistivnega senzorja tlaka so bile anodno zbondirane na rezino Pyrex stekla. Površinski profili obeh rezin pred in po bondiranju so bili posneti s Taylor-Hobson-ovim povrsinskim profilometrom. Vpeljana je preprosta metoda za nedestruktivno oceno bondirnih parametrov in kvalitete bonda, ki je uporabna tudi med samim tehnoloskim procesom. Merilni rezultati na testnih strukturah stanko silicijevo membrano kazejo ukrivljenost membrane po zaključenem bondirnem procesu. Raziskani so bili možni vzroki za takšno obnašanje testnih struktur.

#### **1. Introduction**

Since Wallis and Pomeratz /1/ first introduced anodic or electrostatic bonding in 1969, this technique became one of the basic steps in the fabrication of micro-electro mechanical systems (MEMS). Beside usage of anodic bonding for joining silicon wafer to glass wafer, several related techniques were developed in recent years such as anodic bonding using sputtered glass /2,3/, evaporated glass /4-6/ and spin-on glass /7/. These methods are used for vacuum packaging, hermetic sealing, and encapsulation of MEMS as well as fabrication of reference cavities for pressure and acceleration sensors.



Figure 1: Pyrex wafer bonded to silicon wafer with piezoresistive pressure sensor structures (type2). Pressure sensors structures without diaphragm (type 1) are located at the wafers edge

In this paper, 7740 Corning Pyrex glass wafers were anodically bonded to silicon wafers with pressure sensor structure (Fig.1 ). Among similar commercially available glasses, the main reason to choose 7740 Pyrex glass wafers in our case was their thermal expansion coefficient, well matched with silicon in a wide temperature range. In addition, relative low volume resistivity of 7740 Pyrex glass enables formation of reliable bond already at low applied voltage and temperature that could be essential in many applications. Despite the fact that the optimal solution is to bond silicon wafer directly to glass, silicon oxide as an intermediate layer was introduced in our case to analyze the effects during the bonding of structurized silicon wafers to flat Pyrex glass wafers.

Two different types of test piezoresistive pressure sensor structures fabricated on the same wafer were investigated. The only difference between both is that in the first structure anisotropic etching of diaphragm is not performed. These structures (type 1), located in narrow area at the wafers edge (Fig.1 ), were used as the bond strength test structures based on the blade insertion technique /8-12/ to induce delamination. The second test structures (type2) were used to study fabrication of reference cavity under pressure sensor diaphragm.

### **2. Bonding mechanism**

Bonding mechanism itself is not yet completely understood, but it is generally agreed that bonding is primarily due to the presence of mobile sodium ions in Pyrex glass. At elevated temperatures (yet below the softening point of Pyrex at 821 °C), positive sodium ions are mobile enough for Pyrex to behave like conductor (Fig.2).





When a DC voltage (V) is applied across the silicon-glass sandwich (Fig.3a), sodium ions in glass are transported toward the cathode. The more strongly bounded negative oxygen ions in glass are left in glass adjacent to the silicon surface, forming negative space charge layer. This negative charge layer in glass, together with positive charge in silicon, creates a high electrostatic field across thin air gap between both surfaces. As a consequence, a strong electrostatic pressure pulls both wafers into intimate contact (Fig.3a). This effect can be easily observed during the bonding process on transparent Pyrex glass as the light grey interface between silicon and glass becomes dark grey.



#### Figure 3: Charge and potential distribution during anodic bonding process: a) before and b) after intimate contact between silicon and Pyrex wafer

Once wafers are in intimate contact (Fig.3b), almost all of the applied voltage (V) is dropped across the narrow space charge layer in glass  $(V_q)$ , resulting in extremely high field, strong enough to develop transport of oxygen ions to the bonding surface. As a consequence, irreversible Si-O-Si bonds in the interface, joining Pyrex and silicon, are presumed to occur.

#### **3. Experimental**

Test structures of silicon pressure sensors, fabricated on 3-inch, CZ grown, <100> crystallographic oriented,  $20O$ hmcm, n-type,  $374 \mu$ m thick, double side mechanically polished silicon wafers (details reported elsewhere /13/) were bonded to commercially available 4-inch, 725µm thick 7740 Corning Pyrex glass wafers (Figs.1,4). Before bonding, a thin layer of silicon nitride (70nm) - that covers thin silicon oxide (500nm) on the backside of test structure used for mask during diaphragm etching in KOH - was removed in RIE plasma etcher.

During etching of 20µm thick pressure sensor diaphragms, silicon wafer was placed to a holder that protects wafer front-side from aggressive KOH. Because of the holder sealing, a narrow region at the edge of silicon wafer backside was also protected from KOH etching. This is the reason why diaphragms near wafers edge are not etched (Fig.1 ). This un-etched region was used in our case for the non-destructive anodic bond strength characterization /15,16,17/.



Figure 4: Anodic bonding process setup.



Figure 5: Typical anodic bonding current vs. time.

After surfaces of both wafers were characterized with Taylor-Hobson Talysurf surface profiler, they were cleaned with 01 water and dried with nitrogen. As suggested by Resnik et al. /14/, both wafers were put into an intimate contact in cleanroom ambient at room temperature immediately after surface preparation to avoid particles that cause voids. Silicon-Pyrex structure was bonded together by applying high DC voltage (730V) at temperature 370°C in air atmosphere, using Cimarec hot plate with ceramic top (Fig.4).

Bonding temperature was monitored by thermocouple mounted in aluminium plate. Anodic bonding current was measured during bonding process and a typical result is shown in Fig.5. After bonding process, the surface was scanned again in the same areas as before, using the same Talysurf surface profiler setup.

### **4. Results & discussion**

#### **4.1 Structure without diaphragm (type 1)**

Surface profiles (Figs. 6-11) of test structures without diaphragm from the wafer edge (Fig.1) were scanned with Talysurf in length of 1. 7mm with speed of 0.5mm/s. Profiles were normalized, i.e. rotated till beginning and end of measured curve was in horizontal line.



Figure 6: Surface profile of silicon wafer back-side covered with 500nm thin silicon oxide mask scanned at wafer edge before bonding. Diaphragms of pressure sensors at the wafers edge were not etched.

Before anodic bonding, silicon and Pyrex glass wafer were scanned on both sides (Figs.6-9). In Fig.6, surface profile of silicon wafer back-side is shown. Steps in this profile



Figure 7: Surface profile of silicon wafer front-side scanned at wafer edge before bonding. Peaks represent 500nm thin aluminium metallization lines of single pressure sensor.

(Fig.6) originate in approx. 500nm thick silicon oxide square diaphragm mask (Fig.1), which is used here as anodic bonding strength test structure. Surface profile of silicon wafer front-side (before bonding) in Fig. 7 also contains groups of steps, originating in approx. 500nm thick aluminium metallization lines of pressure sensor. Surface scan of Pyrex glass wafer both sides, front and back, before anodic bonding procedure are presented in Figs.8 and 9, respectively.



Figure 8: Surface profile of Pyrex glass wafer frontside before bonding.



Figure 9: Surface profile of Pyrex glass wafer backside before bonding.



Figure 10: Surface profile of Pyrex glass wafer frontside after bonding.

After silicon and Pyrex wafers were anodically bonded together, with back-sides in contact, surface scans of bonded wafers front-sides were repeated in the same areas as before. Results are presented in Figs.10-11. In both figures, it is clearly seen again the modulated surface profiles with silicon oxide mask as before on Fig.6. Similar amplitudes of modulated surface profile were measured on both surfaces (240µm on Pyrex surface (Fig.10) and 260um on silicon surface (Fig.11)).



Figure 11: Surface profile of silicon wafer front-side after bonding.

Measured results clearly show that both, silicon and Pyrex wafer bend within test structure with silicon oxide mask. Bonding plane lies at 260nm from the interface between silicon and silicon oxide (Fig.12). From several measuring methods, the strength of the bond between two wafers of different material can be evaluated by technique developed on double cantilever cracking under constant wedging condition (Fig.13) /8-12/. In our case, both parameters, blade thickness (2h) and crack propagation (c) were substituted by thickness of thin silicon oxide mask (500nm) and by distance between mask and bonding point of both wafers at the centre of diaphragm (135µm in Fig.12), respectively.



#### Figure 12: Cross-section of bond test structure with measured parameters

In contrast to silicon-silicon wafer bonding, in silicon-Pyrex anodic bonding, the distance c between mask and bond is easily determined by optical microscope. Because bonding within test structure occurs if bond energy is greater



Figure 13: Double cantilever test geometry under wedging condition

than the value determined by blade technique, work of adhesion  $W_{AB}$  (per unit area) can be expressed as /12/:

$$
W_{AB} \ge \frac{3h^2 E_{Si} d_{Si}^3 E_g d_g^3}{2c^4 (E_{Si} d_{Si}^3 + E_g d_g^3)} \approx 1.65 GPa\mu m \tag{1}
$$

where 2h is silicon oxide mask thickness, c is distance between mask and bond,  $d_{Si}$  is thickness of silicon wafer,  $d_g$  is thickness of Pyrex wafer, and  $E_{Si}$  and  $E_g$  are Young's -modulus of silicon and Pyrex, respectively. It is necessary to emphasize that the determined work of adhesion  $W_{AB}$  = 1,65GPaµm, represents a quantitative estimation of bond strength between silicon and Pyrex within the test structure. Therefore, the presented approach based on simple test structure can be used for in-situ non-destructive evaluation of both, anodic bonding process parameters and bond quality, as a comparative method on different parts of a single wafer, between wafers in one run and between different runs as well.

#### 4.2 Structure with diaphragm (type 2)

Test structures with 20um thick diaphragm were characterized in the same manner as structures without diaphragms. Surface profile scans of these structures are presented in Figs.14-17. Despite thin silicon diaphragm, no particular difference was observed in surface profile scan at the centre of silicon wafer front-side before bonding (Fig.14) compared to scan at the edge of silicon wafer shown in Fig. 7. On the other hand, silicon wafer back-side profile in wafers centre before bonding (Fig.15) shows distinctive property of anisotropic etched silicon wafer. Etching depth of 354µm was measured in presented scan.

Compared to structures without diaphragms, no significant differences were obtained from surface scans of Pyrex glass either before or after bonding procedure. This is the reason why those scans are not presented here. Much more interesting results were found in surface scan of silicon wafer front-side after bonding procedure (Figs.16, 17). A magnified section of scanned profile in Fig.16 is presented in Fig.17.

After bonding procedure, deflection peaks in value of 500nm were measured on 20µm thin silicon diaphragms



Figure 14: Surface profile of silicon wafer front-side scanned at wafer centre before bonding. Peaks represent 500nm thin aluminium metallization lines of single pressure sensor.







Figure 16: Surface profile of silicon wafer front-side scanned at wafer centre after bonding shows deflection of pressure sensor diaphragm.

(Figs.16, 17). This result was the reason for a further study of its origins. First, electrostatic pressure involved during anodic bonding procedure was investigated as the possible cause for mentioned deflection. In the first moment,



Figure 17: Zoom of surface profile of silicon wafer front-side scanned at wafer centre after bonding shows deflection of pressure sensor diaphragm.

when a high DC voltage is applied to the silicon-Pyrex structure, value of electrostatic pressure  $p$  under non-deflected diaphragm can be calculated from the following equation /18/:

$$
p = \frac{1}{2} \varepsilon_0 \varepsilon_r \frac{V^2}{h^2}
$$
 (2)

where V represents applied DC voltage (assuming neglectable depth of space charge layer in Fig. 3),  $h$  is the distance between silicon diaphragm and Pyrex glass,  $\varepsilon_0$  is permittivity of free space (the ideal vacuum), and  $\varepsilon_r$  is dielectric constant or relative permittivity of air (1,00059). Due to 354um thick air gap h between silicon diaphragm and Pyrex glass, a value of 18.8Pa was determined for electrostatic pressure that press diaphragm toward glass during anodic bonding procedure. This amount of electrostatic pressure can easily be neglected.

Next, thermally induced mechanical stress as possible cause for diaphragm deflection was studied. As presented elsewhere /19/, thin silicon diaphragm could deflect at elevated temperature due to the mismatch of thermal expansion coefficients of thin layers that covers the diaphragm, despite the fact that the residual mechanical stress in diaphragm at room temperature could be neglected. However, this deflection disappears when such a structure is cooled down to room temperature. This is the reason that thermally induced mechanical stresses were also rejected as the cause for measured diaphragm deflection.

Finally, due to the fact that anodic bonding procedure was done at normal air pressure (10<sup>5</sup>Pa), the main reason for diaphragm deflections, presented in Figs.16 and 17 was found in the well known gas equation:

$$
\frac{p_1 V_1}{T_1} = \frac{p_2 V_2}{T_2} = const.
$$
 (3)

where  $p_1$  represents air pressure,  $V_1$  is reference cavity volume during anodic bonding procedure at elevated temperature  $T_1$ , while  $p_2$  means pressure (vacuum) under thin diaphragm in reference cavity with volume  $V_2$  at room temperature  $T_2$ . Compared to reference cavity volume  $V_1$ , change of cavity volume  $V_2$  due to the deflected diaphragm is neglectable in our case. Because both wafers, silicon and Pyrex, came into intimate (hermetic) contact at air pressure and temperature 370°C immediately after DC voltage was applied, pressure (vacuum) in reference cavity *P2* in value of 6.49kPa was determined from Eq.3. The same diaphragm deflection can be achieved, if pressure of 93,51 kPa is applied on the diaphragm from above.

Using our laboratory computer simulator for thin silicon diaphragm deflections /20/, pressure above 500nm deflected thin silicon diaphragm was determined. Simulations for piezoresistive pressure sensor structure deflection show that such a diaphragm deflects for 500nm (maximal deflection in centre of diaphragm) in case when pressure of 77.88kPa is applied to its top surface.

Simulated result (77.88kPa) does not match with the result obtained from gas equation (93.51 kPa). The reason for that could be oxygen generation during anodic bonding procedure, an assumption widely accepted in the literature /21,22/.

### **5. Conclusion**

Test structures, based on piezoresistive pressure sensors were anodic bonded to Pyrex glass and analyzed. Surface profiles of both, silicon and Pyrex wafer, were measured before and after bonding process, using Taylor-Hobson Talysurf surface profiler. A simple test method for non-destructive in-situ evaluation of both, anodic bonding parameters and bond quality, was introduced. Anodic bond strength between 7740 Corning Pyrex glass wafer and silicon wafer was determined. Measured results on test structure without diaphragm unambiguously show bending of both, silicon and Pyrex wafer. Bond strength within test structures was evaluated by a new technique developed on double cantilever cracking under constant wedging condition. The proposed approach is appropriate for a simple, efficient quality control in anodic bonding process. Measured results on test structures with thin silicon diaphragm show diaphragms deflection after the anodic bonding process was performed. Possible causes for such behaviour were analyzed and discussed.

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