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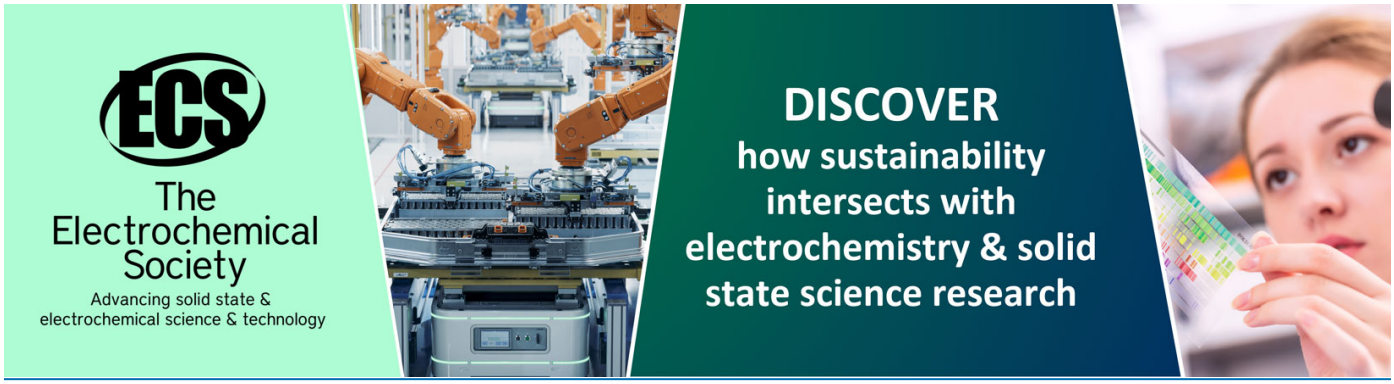

J. Micromech. Microeng. 5 (1995) 98–102. Printed in the UK

Characterization of the electrostatic bonding of silicon and Pyrex glass

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Received 14 December 1994, accepted for publication 3 January 1995

Abstract. The electrostatic bonding of silicon and Pyrex glass was studied in order to find out the influence of process parameters on the final result and to be able to optimize the process with regard to the fabrication of silicon sensors. The most important parameters are the temperature and the voltage. The main criterion used for the optimization of their values was the induced stress in the silicon part of the bonded ensemble. It was found that a temperature of 360 °C and voltages in the range 750–1000 V are suitable.

1. Introduction

The electrostatic bonding of silicon and glass has become one of the important steps in the fabrication of microsensors. Although it seems to be a simple process, a good understanding of the factors that influence the final result is mandatory to create good and reproducible devices. The anodic bonding is supposed to provide a strong, hermetic seal which protects the silicon chip from the environment. It also makes possible the fabrication of reference cavities for pressure and acceleration sensors, as seen in figure 1 [1–3]. However, the internal stress due to the thermal mismatch between the glass and silicon often negatively affects the devices' specifications. As an example, the compressive stress induced by the anodic bonding in the silicon wafer can easily generate buckling for clamped structures such as membranes and resonant beams.

The purpose of this work was to establish these influences and to optimize the process, based on quantification of the mechanical strength in the bonded structure and the thermal residual stress. In the case of piezoresistive or resonant sensors the transduction phenomenon is based on the dependence of some electrical properties (resistivity and resonance frequency, respectively) on the stress in the structure. The thermal zero stability is strongly influenced by the stress induced by the packaging. For capacitive sensors, tight control of the distance between the two electrodes is mandatory for a reproducible device. Built in stress in a membrane, however, can negatively affect the device's performance: if e.g. a large compressive stress is present, buckling will fully deteriorate its functionality. Hence, whatever the transduction principle is, the thermal stress is one of the most important specifications of an anodic bond in micromachined mechanical sensors.

The materials used in the experiments were p-type silicon wafer, 400 μm thick, and #7740 Corning Pyrex glass wafers, 1500 and 500 μm thick. The bonding test were performed using square-shaped silicon and glass pieces with an area of 1 cm^2. The silicon wafers were either flat, with no pattern on them, or with MESA structures as shown in figure 2.

The chosen methodology consisted of monitoring the current during the anodic bonding, and measuring the strength of the bond and the induced stress in silicon.

2. Process parameters

The investigated parameters of the process are the applied DC voltage over the wafers, the bonding temperature, the pressure, the gas composition, the thickness of the glass wafers and the pre-treatment of the silicon surface.

Temperature plays an important role in the anodic bonding process since it increases the ion mobility in glass. As the glass temperature is raised, the resistivity decreases exponentially and a rapid build up of the space charge occurs at the interface, giving rise to electrostatic forces which pull the two wafers into intimate contact [4, 5]. Some typical current–time curves during bonding at different temperatures are shown in figure 3(a). Similar characteristics can be obtained for processes with the voltage as the (figure 3(b)) parameter. The higher the temperature or the voltage the higher the peak current and the smaller the time required for a complete bonding. The magnitude of the current peak gives information about the strength of the bond.

The bonding is achieved much faster in air at atmospheric pressure, than in vacuum (see figures 3(c) and 3(d)). This fact supports the assumption that the free oxygen from air helps the anodic bonding to occur. However, this cannot be the only explanation for the worse

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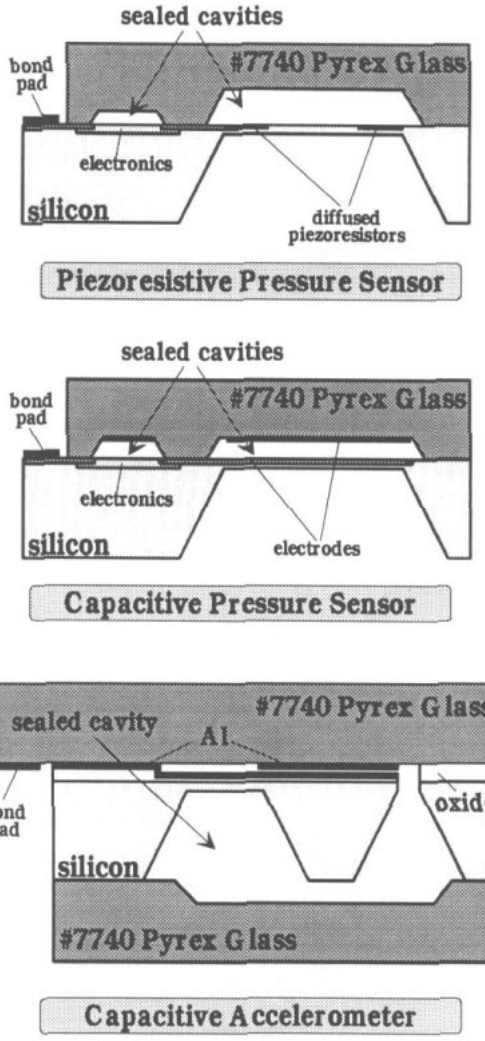

Figure 1. Some applications of the anodic bonding in the fabrication of micromachined sensors.

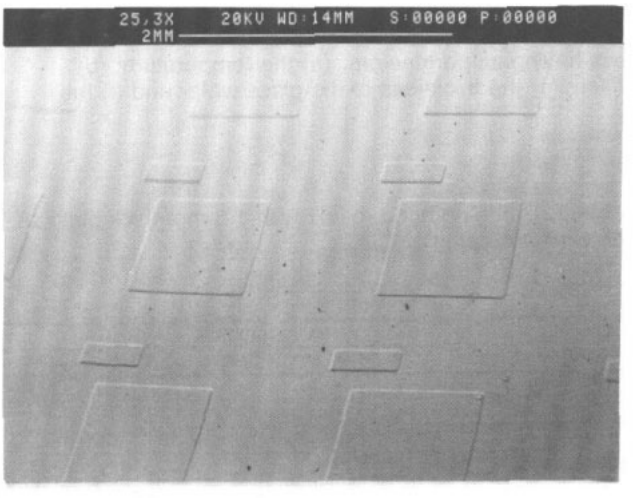

Figure 2. A SEM picture showing the MESA structures that have been used in some of the experiments.

behaviour in vacuum. A possible reason could be the poor heat transfer in vacuum.

The glass thickness, as expected, has a great influence in the bonding process. A thicker glass means a higher electrical resistance, which decreases the effective applied voltage to the glass/silicon interface, and thus generates a weaker bond (see figure 3(c)). It also induces higher stresses in the silicon wafer, as will be shown below.

The anodic bonding process could be explained by the formation of Si–O–Si bonds originating either from silicon oxidation at the interface or by thermodehydration of the silicon–glass ensemble. The formation of a SiO_2 layer between silicon and glass during bonding was proved by backscattering spectroscopy [6]. Experiments carried out in different ambient conditions showed that the success of the bonding process depends on the availability of oxygen at the silicon–glass interface. Hence, silicon oxidation helps in the formation of the anodic bonding.

In order to get some information about the process of thermodehydration of the silicon–glass ensemble, experiments were carried out using either hydrophilic or hydrophobic surfaces. Boiling in 65% nitric acid was used to achieve a hydrophilic surface. Dipping in buffer HF removes the native oxide and leaves the surface hydrophobic.

Some current curves obtained during the anodic bonding are presented in figure 3(f). As can be seen, at a higher temperature there are no major differences generated by the pre-treatment of the wafers. Hydrophilic silica surfaces contain a large number of −OH groups which can form hydrogen bonds when contacting two wafers. More hydroxyl groups at the surface means more available bonding sites. At temperatures around 200 °C the −OH starts to dehydrate and the hydrogen bonds are replaced by Si–O–Si bonds. After an HF dip, the silicon surface consists mainly of Si–H bonds which are chemically stable, and there are few −OH groups present. Hence, the bond failed (see next section). However, at temperatures higher than 400 °C strong Si–Si bonds can be formed through hydrogen desorption [7] and the bond strength is improved.

In conclusion, a higher bonding temperature provided a better seal quality. The bonding is performed easily and the number of non-bonded regions is smaller compared to a low bonding temperature. The step coverage capability of the glass is improved and, hence, the size of the non-bonded area around the trapped foreign particles at the interface and around metal paths is also smaller. However, the induced stress is larger with increasing bonding temperatures as will be shown by the experiments presented below.

3. The bond strength

The mechanical strength of the bond was measured by using a tensile test. The chips were loaded gradually until the bond broke apart. In tables 1 and 2 the bond/no bond matrix is presented for samples bonded in air at 1 atmosphere and high vacuum, respectively, using flat silicon wafers and 1.5 mm thick glass. The white, light grey and dark grey cells represent respectively, non-bonded, partially bonded, and completely bonded chips. Inside the cells the value of the measured fracture stress by the tensile test is written. No stress measurement was performed for the empty cells.

In table 3, the measured bond strengths for hydrophilic and hydrophobic surfaces and two temperatures are presented. This time MESA structures were present on the silicon wafer and the glass wafer thickness was 0.5 mm.

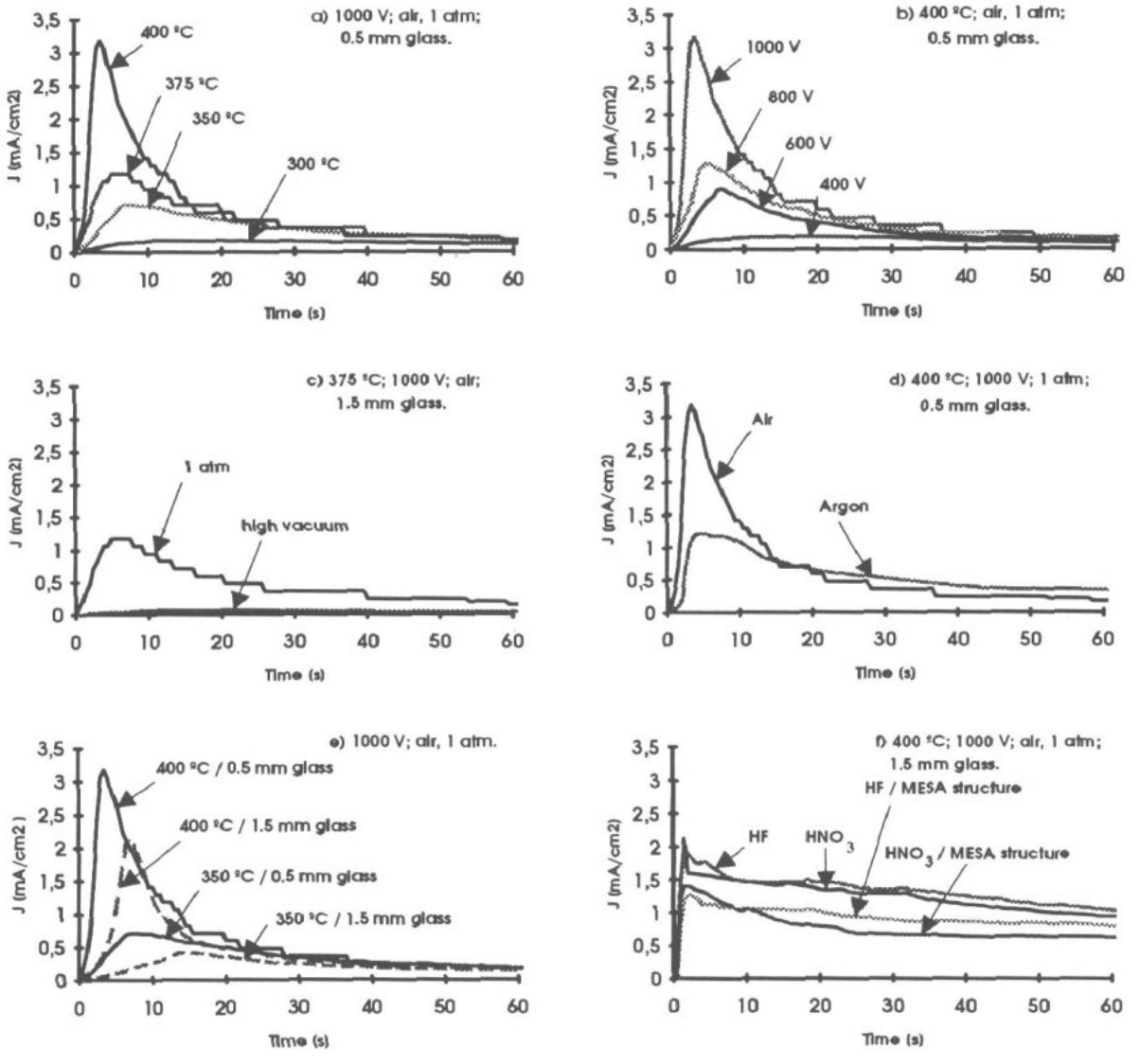

Figure 3. Current characteristics of the anodic bonding process having as parameters: (a) the temperature; (b) the applied voltage; (c) the gas pressure; (d) the gas composition; (e) the thickness of the glass wafer and (f) the pre-treatment of the silicon surface.

Table 1. Bond–no bond matrix with 1.5 mm thick glass at 1 atm in air.

DC VOLTAGE	TEMPERATURE					
	300 °C	350 °C	375 °C	400 °C	425 °C	450 °C
175 V	-	-		1.1 MPa	3.3 MPa	3.4 MPa
350 V	-	1.0 MPa	1.8 MPa	2.0 MPa	3.5 MPa	3.6 MPa
530 V	1.2 MPa	1.95 MPa	2.4 MPa	2.8 MPa	3.65 MPa	3.8 MPa
700 V	1.5 MPa	2.2 MPa	2.75 MPa	2.8 MPa	3.5 MPa	3.75 MPa
880 V					3.3 MPa	
1050 V					3.75 MPa	

Table 2. Bond–no bond matrix with 1.5 mm thick glass at 3.5×10^{-5} mbar in air.

DC VOLTAGE	TEMPERATURE					
	300 °C	350 °C	375 °C	400 °C	425 °C	450 °C
175 V	-	-	-	-	1.2 MPa	1.4 MPa
350 V	-	-	0.9 MPa	1.2 MPa	2.45 MPa	2.4 MPa
530 V	-	-	1.9 MPa	2.1 MPa	2.4 MPa	2.6 MPa
700 V	-	1.8 MPa	2.0 MPa	2.2 MPa	2.3 MPa	2.5 MPa
880 V	-				2.4 MPa	
1050 V					2.5 MPa	

As can be seen, the bond strength generally increases with the applied voltage and temperature. It is also higher for bondings performed at higher pressures and for hydrophilic surfaces.

The bond strength was found to be satisfactory for temperatures in the range 350–400 °C and voltages of 500–1000 V. Hence, the further optimization of the process was done with regard to the residual stress.

4. The thermal residual stress

The thermal residual stress generated by the anodic bonding process was measured either via the change in the curvature of the wafers or by Raman spectroscopy.

Table 3. Strength of the bond with 0.5 mm thick glass at 1 atm in air.

TYPE OF THE SURFACE	BONDING TEMPERATURE	
	300 °C	400 °C
hydrophilic	2.5 MPa	12.4 MPa
hydrophobic	-	10.8 MPa

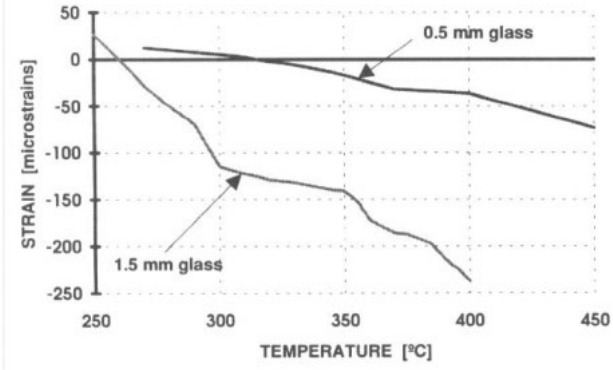

Figure 4. The induced strain in the silicon at the bond interface. Parameters: 1000 V, air at 1 atm.

The influence of the bonding temperature on the bending and thermal stress in the two materials was determined by measuring the difference between the curvature of silicon and glass wafers before and after bonding. The residual stress in silicon was then computed. Figure 4 shows the relation between the strain in silicon at the interface and the bonding temperature, for glass samples with 0.5 or 1.5 mm thickness.

As expected, the thicker the glass wafer the higher the induced stress in silicon. These curves are in good agreement with the data presented in [8] and theoretical calculations from [9].

It can be easily seen that there is a temperature which provides a no-stress bond. For 0.5 mm glass, that temperature is 315 °C, while for 1.5 mm glass it is lower (~260 °C). The above mentioned temperatures should only be considered as estimates because the thermal expansion coefficients may vary from batch to batch. Anyway, it is clear that a high process temperature induces a higher tensile stress. The above presented results are valid for the case of a silicon wafer without cavities. In practice, the thickness of the silicon is not constant over the device and the thinner part will be submitted to a higher level of the residual stress. Therefore, the thermal residual stress can become critical as the size of the devices becomes very small, as for example, where membranes are located.

Aside from the presented thermal considerations, there are some other stress-inducing effects which contribute to the compressive component in silicon [9]: temperature gradients in the silicon and/or glass during bonding; self-heating due to resistive dissipation of the current during anodic bonding; glass composition gradients from Na^+ electromigration. The initial curvature of the wafers also influences the value and/or the results of the stress induced in silicon.

In figure 5 the induced stress in silicon measured by Raman spectroscopy is presented for different batches. The

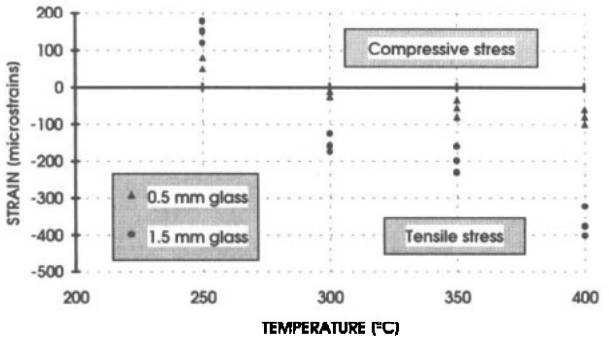

Figure 5. The strain measured by Raman spectroscopy. Parameters: 1000 V, air at 1 atm.

results are similar to those presented in figure 5. The only big difference consists in the higher values of the stress obtained by this method. The difference can be explained by the fact that by Raman spectroscopy the total strain is measured, whereas in the bending method only the strain induced by the anodic bonding process was computed.

In the case of capacitive sensors, silicon under compression must be avoided because it easily leads to buckling of thin membranes or other multiple-clamped structures. Since the distance between the two electrodes is very small (1–2 μm or even smaller), buckling may easily cause the total failure of the device. Since tensile stress only reduces the mechanical response and introduces a soft non-linearity [9], silicon under tension is preferable. In order to make sure that buckling will not occur, a sufficiently large margin must be provided towards higher temperature because the thermal properties of glass may vary considerably from batch to batch [8]. Temperatures above 360 °C are thus recommended. For piezoresistive and resonant sensors, the stress must be reduced as much as possible, regardless whether it is compressive or tensile.

It should be pointed out that the particular design and functions of the final device influence the choice of the temperature and the applied voltage. For example the thickness and density of the metal paths, the presence of a silicon oxide on the silicon wafer are also important parameters. We found that metal layers thicker than 200 nm prevent intimate contact between the two wafers for the lower sealing temperatures [6, 9]. The non-bonded area around them increases with decreasing bonding temperature. The SiO_2 should not exceed 300 nm for a good seal quality [6]. As far as the voltage is concerned, it must be high enough to provide strong electrostatic forces able to bring the two surfaces into intimate contact. Higher voltages allow bonding with lower temperatures and improve the step-coverage capability. However, increasing the bias voltage increases the risk of buckling because it increases the compressive stress due to the Na^+ ion migration. Values of 750–1000 V are suitable.

The large electrostatic forces developed during anodic bonding act on any mobile structures such as diaphragms, beams, suspended masses, and attract them towards the glass wafer. These structures could remain stuck on the glass even after the voltage is removed. At 400 °C, sintering of aluminium occurs and movable structures can remain permanently attached to the glass. A solution to this

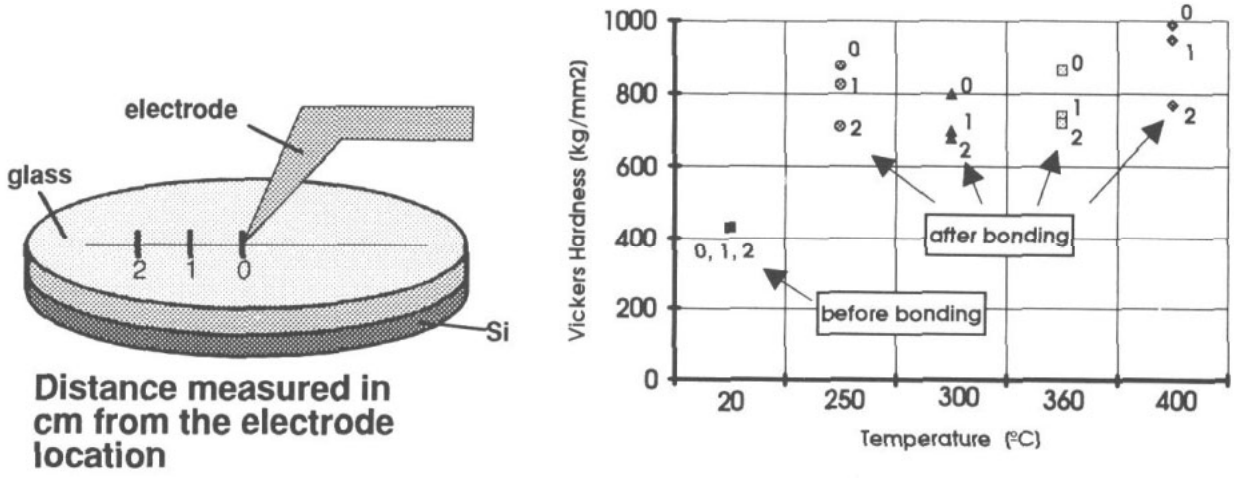

Figure 6. The Vickers Hardness of the glass before and after bonding at 1000 V, in air at 1 atm. The values were measured at different distances from the electrode.

problem is to decrease the process temperature. It is to be mentioned that the decrease of the bonding temperature requires an increase of the applied voltage, especially when bonding the second glass wafer of a glass/silicon/glass stack. Bonding at 360 °C was performed with good results [3]. The use of another metallization system can help overcome the sticking of the flexible structures (Ti/Pt for example). A Si_3N_4 layer deposited on the beams avoids the sticking too.

5. Hardness

It was observed that the dicing of the silicon–glass ensemble was much more difficult than the dicing of an unprocessed glass wafer. This has led to the conclusion that the glass hardens during the anodic bonding. The Vickers hardness of the glass was measured before and after bonding.

The results are shown in figure 6. For each temperature, the highest value was obtained at the point where the electrode was in contact with the glass. The lower values correspond to regions located at 1–2 cm from the electrode. The glass hardness after bonding is up to two times higher than before the process. Although the values obtained on samples bonded at 400 °C are somewhat higher than those corresponding to lower temperatures, the differences are not relevant. The change in the hardness of the glass is generated mainly due to the migration of the sodium ions towards the electrode and their accumulation in the nearby region. Therefore, it is recommended to use a point electrode in order not to increase this effect unnecessarily over the whole wafer. Moreover, this type of electrode was found to be the best at avoiding gas-trapping during bonding.

6. Conclusions

A proper analysis of the actual situation is required for an optimal choice of the parameters of an electrostatic bonding process. The geometry and the functions of the device are important factors that must be taken into account. The main criterion for the process parameters is the stress induced in the silicon part of the bonded ensemble. Although high temperatures and voltages are beneficial for the bond quality, they can induce a residual stress that deteriorates device performance.

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