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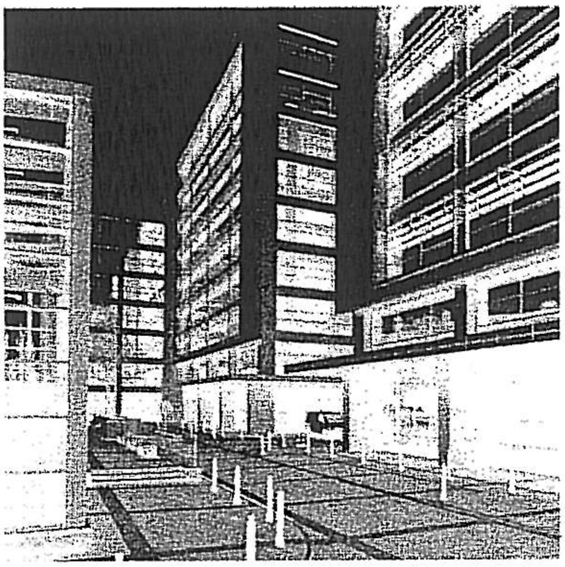

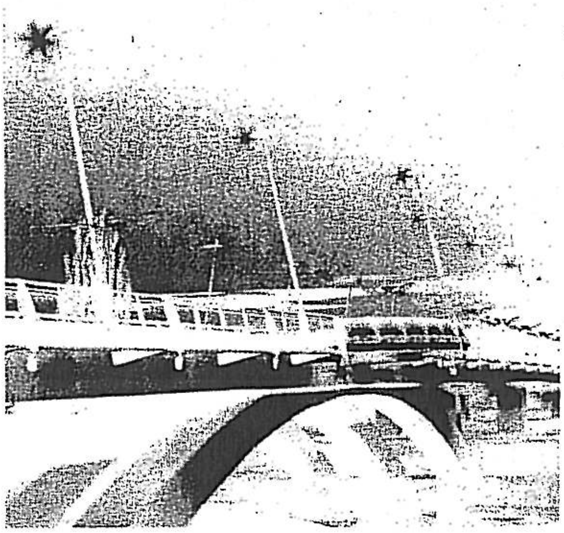

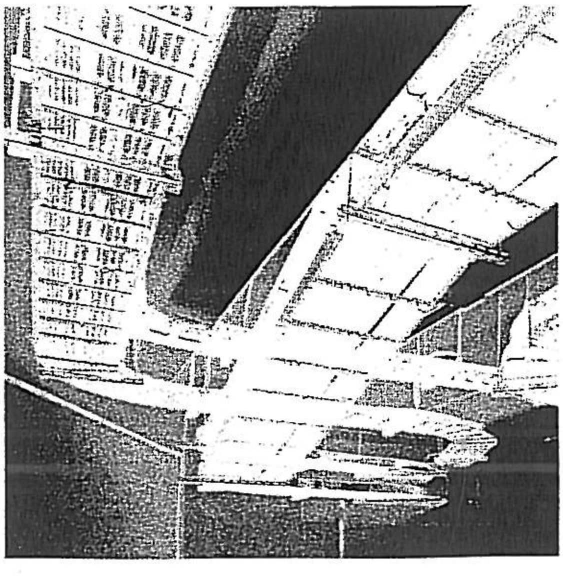

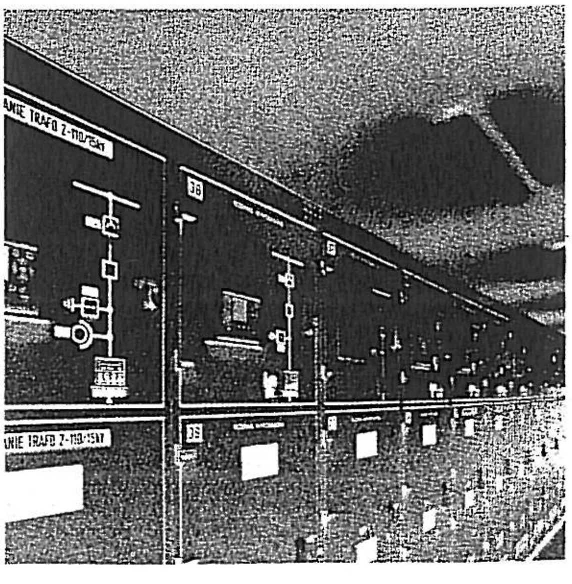

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Bonding of silicon and crystal quartz wafers at the minimized residual stresses

Abstract- *In this work, strong low-temperature bonding of silicon and crystalline quartz wafers, effecting in mechanical strength, which is close to initial materials has been described. High bonding strength is associated with minimization of the residual stresses, optimization of surface activation, and application of an electric field during annealing. The bonding has a wide application field because both, silicon and crystalline quartz are key materials for many devices including generators, high frequency filters, gyroscopes, microbalances of high stability, etc.*

Streszczenie- *W pracy przedstawiono proces uzyskiwania silnych połączeń spajalnych płytek kwarcowych i silikonowych w niskiej temperaturze. Mechaniczna wytrzymałość połączeń jest bliska wytrzymałości materiałów wyjściowych. Wysoka wytrzymałość połączenia wynika z minimalizacji naprężeń własnych materiału, z optymalnego procesu aktywacji powierzchni płytek oraz z zastosowania pola elektrycznego podczas procesu wygrzewania. Połaczenia tego typu mogą być szeroko stosowane gdyż zarówno kwarc krystaliczny jak i silikon wykorzystywany jest do wyrobu szeregu urządzeń; w tym, w mikro i nanotechnologiach.* ***(Proces uzyskiwania silnych połączeń spajalnych płytek kwarcowych i silikonowych w niskiej temperaturze)***

Keywords: Bonding, crystal quartz, silicon, residual stress, plasma surface processing
Keywords: Połączenia spajalne , kwarc krystaliczny, silicon, naprężenia własne, plazmowa obróbka powierzchni

Introduction

Direct bonding of silicon and quartz wafers can be applied to the fabrication of advanced micro-electromechanical and optical devices. Conventional bonding technology of identical materials, such as quartz-quartz, silicon-silicon, etc., is well known and typically involves an annealing at elevated temperature. High temperature increases the mobility of atoms across the interface that largely determines a bonding strength. When bonded structure consists of materials with different thermal expansion coefficients excessive internal stresses may arise at the interface as a result of high annealing temperature. Moreover, preprocessed wafers should not be exposed to high temperature in order to avoid the damage of the structures. Therefore, the development of a low-temperature technology is a key requirement of the strong bonding of dissimilar materials such as silicon and quartz pair. An alternative to high-temperature annealing can be the through preparation of the surfaces for each specific pair of dissimilar materials. The most promising results are achieved when the surface preparation includes a plasma treatment step. Plasma treatment has many applications [1, 2] including general decontamination, broad spectrum of surface modification techniques, production of new materials, displays, etc. Plasma causes wide range of effects and significantly changes the structure of the surface. Charged particles can ionize atoms, disrupt the crystal bonds, and give rise to dangling bonds formation. Plasma creates surface charges through implantation of ions, forms the electric fields, which in turn, increase the chemical activity of the crystal surface and affect the ion mobility. These and similar effects form the concept of plasma surface activation and their influence on bonding strength is widely discussed. A relatively strong bonding of plasma activated surfaces has been achieved in [3-5]. Even such dissimilar materials as crystalline silicon and lithium niobate show strong bonding as result of surface activation [6]. The latter proves that plasma surface treatment has a great potential for MEMS technology. Low-temperature technology can essentially reduce the residual stresses, but does not completely eliminate them for materials with different thermal expansion coefficients. Operating conditions of MEMS (Micro Electro-Mechanical Systems) device include a temperature range, which should be as wide, as possible. Thus, internal stresses distribution must be given proper consideration in the bonded structure design. This work aims to manufacture a strong bonding of silicon-quartz structures with the lowest residual stresses possible. The residual stresses were calculated theoretically. The experiment was performed by plasma-assisted activation of silicon and quartz surfaces, with further annealing in the electric field. Strong bonding close to the mechanical strength of the initial materials has been achieved.

Residual stresses in bi-layer system

The calculations of elastic deformation of bi-layer systems are mostly based on old Stoney's [7] and Timoshenko's [8] approaches although the modern theories progress [9]. Stoney analyzed the model of a thin film deposited on thick substrate. Timoshenko's approach looks the most appropriate for the bonding because it imposes no restrictions on the thickness of the layers. This model was originally developed for analysis of operation of bi-metal strip thermostat and based on the use of the radius of curvature ρ of a structure, which is curved as result of a difference $\Delta\alpha=\alpha_1-\alpha_2$ of the thermal expansion coefficients α_1 and α_2 of the layers. The model is also appropriate for description of the residual stresses during bonding of the plates of dissimilar material at elevated temperature because the bonded wafers usually have comparable thicknesses in the range between 0.1 mm and 1 mm. In the case of the bonding, ΔT means a difference between annealing temperature and room temperature, or more precisely, concrete operating temperature of the bonded structure.

Let h_1 and h_2 be the thicknesses of bonded plates, E_1 and E_2 are their Young's modulus, and ΔT is the difference between annealing temperature and operating temperature of the bonded structure. Then the radius of curvature of the strip of unit width will be [8]

$$\rho = \frac{h}{2\Delta\alpha\Delta T} + \frac{(E_1h_1^3 + E_2h_2^3)(1/E_1h_1 + 1/E_2h_2)/6(h_1 + h_2)}{\Delta\alpha\Delta T} \quad (1)$$

Using (1), the residual stresses can be calculated according to the condition that, on the interface, the unit elongation occurring in the longitudinal fibers of both materials must be equal [8].

(2) $$\alpha_1 \Delta T - \frac{P}{E_1 h_1} - \frac{h_1}{2\rho} = \alpha_2 \Delta T + \frac{P}{E_2 h_2} + \frac{h_2}{2\rho}$$

The first term on the left side of equation (2) represents the elongation of the layer 1 due to thermal expansion, the second term is due to axial force P_1, and the third term is due to bending moment M_1. The right side of equations (2) contains similar terms in regarding to the layer 2. In Fig. 1, the tensile stresses are considered positive (arrows point to the right), compressive stresses are considered negative (arrows point to the left). In the absence of external forces, all forces acting over any cross-section of bi-layer system must be in equilibrium, therefore,

$$P_1 = P_2 = P,$$

$$P(h_1+h_2)/2 = M_1 + M_2.$$

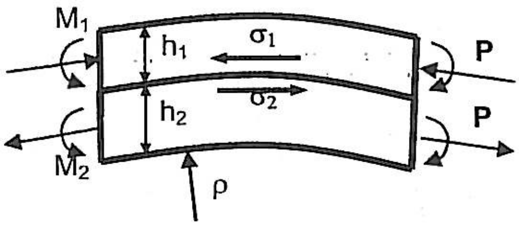

Fig. 1. Schematic diagram of bi-layer structure bended under the action of residual stresses.

The stresses σ_1 and σ_2 immediately adjacent to the interface as viewed from the quartz and silicon, respectively (Fig. 1) are the most important because they directly affect the bonding strength. By applying Hook's Law, the stresses σ_1 and σ_2 could be obtained from (1) and (2),

(3) $$\sigma_1 = -\frac{E_1^* E_2^* h_2 (\Delta\alpha\Delta T - h/2\rho)}{E_1^* h_1 + E_2^* h_2} - \frac{E_1^* h_1}{2\rho}$$

(4) $$\sigma_2 = \frac{E_1^* E_2^* h_2 (\Delta\alpha\Delta T - h/2\rho)}{E_1^* h_1 + E_2^* h_2} + \frac{E_1^* h_1}{2\rho}$$

Equations (3, 4) were deduced for a plate structure bended into a spherical form, therefore $E_1^* = E_1/(1-\nu_1)$ and $E_2^* = E_2/(1-\nu_2)$ are substituted instead of E_1 and E_2 where ν_1 and ν_2 denotes Poisson's ratios for materials of layer 1 and 2 accordingly [10]. In this case, the curvatures in any two orthogonal directions will be equal and constant over the surface of the plate.

For "symmetrical" case of $E^*_1=E^*_2$, the equations similar to (3, 4) are obtained in [10]. The difference between equation (3, 4) and those from [10] is noteworthy. Fig. 2 shows the stresses on both sides of the interface as a function of normalized thickness. If $E^*_1=E^*_2$, the σ_1 and σ_2 individually depend on the thicknesses ratio of the layers and these dependences are non-monotonic functions (Fig. 2(a)). The stress in layer 1 is maximum for thin layer and tends to zero when $h_1/(h_1+h_2)$->1. The same, but "symmetric" dependence takes place for layer 2. As for combined stress $|\sigma_1-\sigma_2|$ at the interface, this stress does not depend on the thickness (Fig. 2(a), straight dashed line) and is always equal to the maximum value of σ_1 or σ_2.

In the general case ($E^*_1 \neq E^*_2$) the "symmetry" is broken, though the individual features of σ_1 and σ_2 are still similar to "symmetrical" case ($E^*_1=E^*_2$). On the contrary to the previous case, the combined stress $|\sigma_1-\sigma_2|$ becomes dependent on the thickness, (Fig. 2,b).

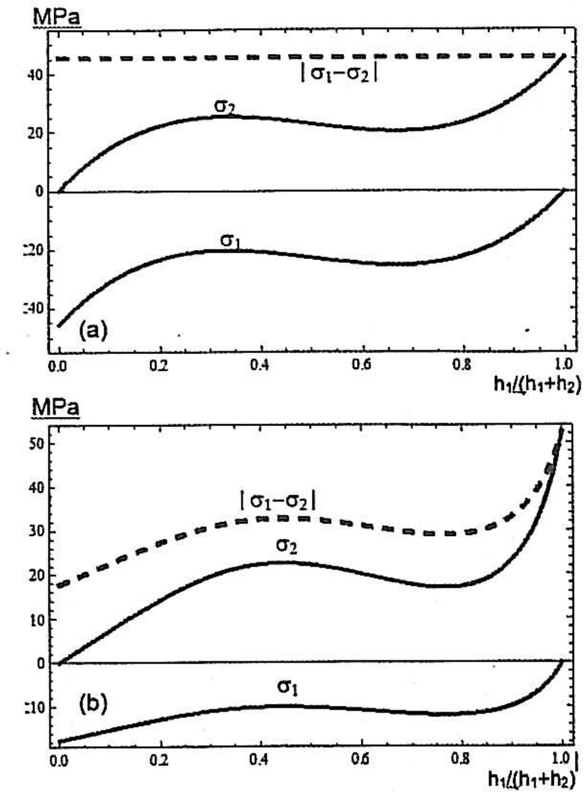

Fig. 2. (a)- residual stresses at the interface of bi-layer structure for $E^*_1=E^*_2=456$ GPa, $\Delta\alpha\,\Delta T=10^{-5}/^0C$ as a function of normalized thickness $h_1/(h_1+h_2)$; (b)- residual stresses at the interface of quartz/silicon structure for $\Delta T=100^oC$, $E^*_1=86.4$ GPa, $E^*_2=256.9$ GPa, $\nu_1=0.17$, $\nu_2=0.28$, $\Delta\alpha=2.05\cdot 10^{-6}/^0C$ as a function of normalized thickness $h_1/(h_1+h_2)$.

The combined stress decreases as $h_1/(h_1+h_2)$ decreases. This tendency becomes more obvious as ratio E^*_1 / E^*_2 tends to diminish. The latter gives an additional opportunity to reduce the residual stresses. For example, if h_1/h_2 =0,2, the residual stresses on the interface of quartz/silicon pair will be approximately 20% lower than in the case when both wafers are of equal thickness. This opportunity takes place exclusively due to the inequality of E^*_1 and E^*_2, as in the case of silicon/quartz and many other pairs.

Experimental

In the experiment, silicon wafers 0.5 mm thick and crystal quartz wafers 0.1 mm thick were used in accordance with previous calculation result ($h_1/(h_1+h_2)$ =1/6). Prior to plasma activation, the silicon wafers were cleaned in two stages. In the first stage, organic contaminations were removed successively with acetone, isopropyl alcohol, and in an ultrasound bath with water. Afterwards, the wafers were dried in N_2 gas. The quartz wafers were cleaned in a mixture H_2SO_4: H_2O_2 = 3:1 at 110°C, in distillated water at 80°C, rinsed in DI water, and dried in N_2 gas. Plasma exposure of both silicon and crystalline quartz plates was realized in a reactive ion etcher (SAMCO RIE-10NR) with the 200W maximum power RF generator. Oxygen plasma was chosen because of its effectiveness in the activation of the surface [10]. Immediately after plasma exposure, Si and quartz wafers were brought into contact, then clamped between two stainless steel plates with well-polished surfaces and placed in a heater for annealing. Identically prepared samples were annealed under pressure of about 25 KPa and temperature 130°C during 8 hours. In addition, DC voltage of about 300V was imposed across the specimen during annealing, as is shown in Fig. 3.

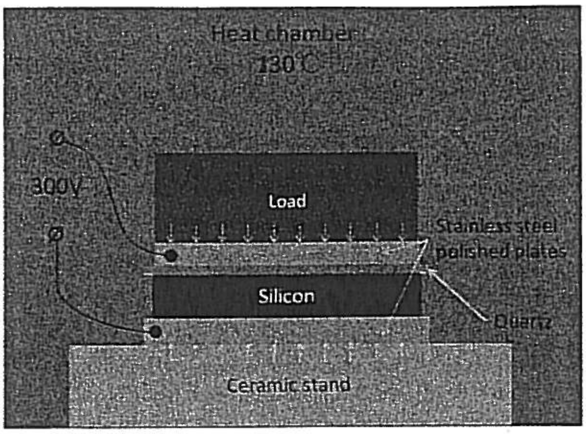

Fig.3. Schematic drawing of the setup for annealing in electric field.

After annealing, the bonded pairs were diced into 12x12 mm pieces for tensile strength measurements. To produce a specimen for the tensile test, two socles were glued to both sides of the bonded pair by epoxy resin. Prior to gluing, socle surfaces were sand blasted and cleaned in an ultrasonic bath with acetone. The schematic diagram of the tensile test is shown in Fig.4. The samples were loaded gradually until they broke.

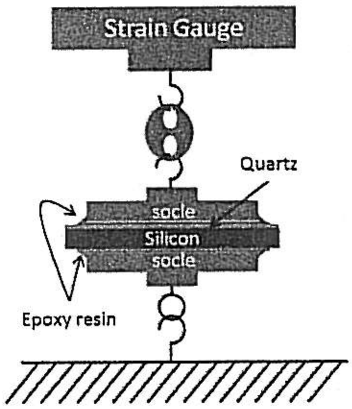

Fig. 4. Schematic draving of tensile test.

Result and discussions

The bonding process consisted of the following successive stages: cleaning of the wafer, surface plasma activation, connecting the surfaces at room temperature and annealing. Taking into account [11], an attempt was made to rinse the wafers with DI water immediately after the plasma activation. However, this rinsing drastically reduced bonding strength in our experiment. Therefore, the rinsing was excluded from the bonding process in the following experiment. A relatively short plasma exposure (15 sec) was used for both, silicon and quartz to avoid a roughening of the wafer surfaces [4] Next, the influence of etching regimes was examined: e. g. flow rate and the pressure of the oxygen inside of ion chamber. It has been found that bonding strength is not changed in the 40 to 150 ml/s range of oxygen flows in the plasma chamber. The oxygen pressure affects the bonding more significantly than the flow rate (Fig. 5). The strongest bonding can be achieved in a narrow interval of oxygen pressure at approximately 5Pa. Moreover, testing shows that the samples activated at this plasma pressure keep bonding when the epoxy glue brakes.

In addition to conventional technology of silicon and quartz bonding, the electric field was utilized during the course of annealing.

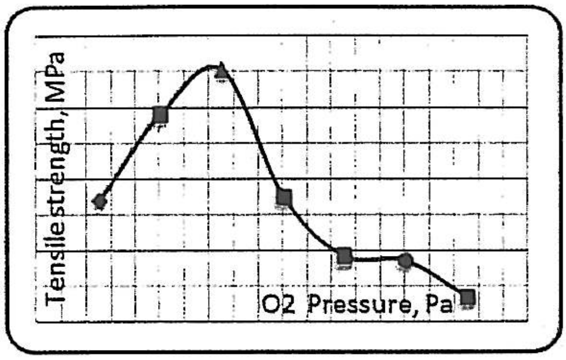

Fig. 5. Tensile strength of silicon/crystal quartz sample as function of O2 pressure inside of chamber during plasma activation

Annealing in the electric field (anodic bonding) provides a very high bonding strength, close to the mechanical strength of the initial bulk materials. Anodic bonding efficiency is associated with the high mobility of alkali ions at high temperature. The movement of ions is controlled by an electric field. However alkali atoms are absent in silicon and quartz wafers. High temperature is unacceptable because it causes an internal stress in the bonded materials with a significant difference in the thermal expansion coefficient. Performed experiment verified that the electric field had a favorable effect on the bonding strength. This result could be due to the fact that the surface, immediately after activation by plasma, represents a loose structure with weak or even broken interatomic bonds. Therefore, the atoms become highly mobile and actively react in the electric field. And vice versa, when the bonds are intensified as a result of an external action, the reaction of the atoms in the electric field becomes weaker. The latter explains the negative role of rinsing the specimen after plasma activation, as an exposure, which saturates the interatomic bonds.

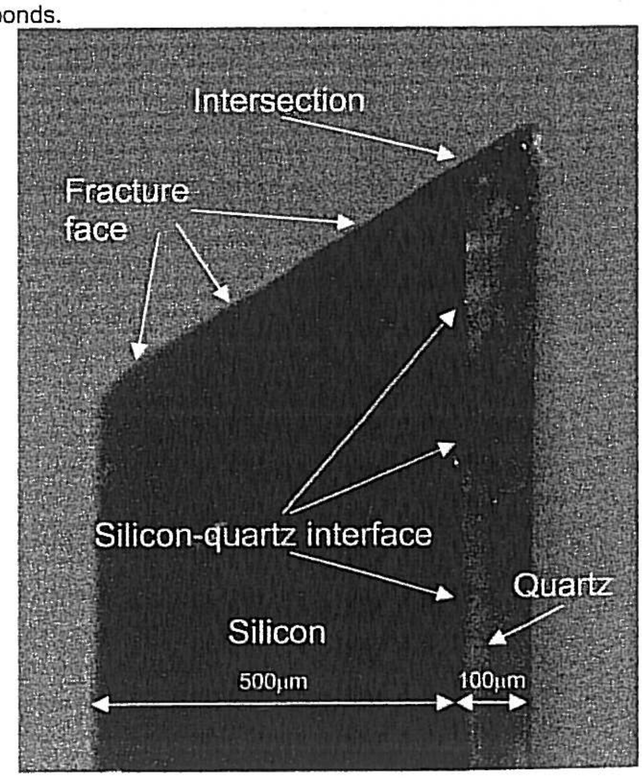

Fig. 6. Fracture face intersects the bonding plane (silicon-quartz interface).

The bonding strength was estimated by the tensile test. The most common crack opening method cannot be used because of inability to separate the bonded wafers by razor. The tensile test showed that the samples mainly kept bonding up to 35 MPa when epoxy glue broke. This result, as well as the fact that it was not possible to separate the bonded wafers by razor, show the strength higher than that achieved at room temperature bonding [3,4,6,11] and even after 300°C annealing [12]. An additional test to qualitatively compare the bonding strength and strength of the bulk material was performed. The samples were specially cleaved and a fracture surface was examined using microscope. As it is seen in Fig. 6, the fracture surface intersects the silicon and quartz crystals and does not include a bonding interface plane. The cleaved surface passes the bonded sandwich structure as it would be a homogeneous bulk medium. That proves that the bonding is of a high strength, close enough to that of the initial bulk material.

Conclusions

In this work, a strong low-temperature plasma assisted bonding of crystalline quartz/silicon wafers has been developed. High bonding strength, which is close to the mechanical strength of the starting materials was achieved through optimization of plasma activation mode, minimize the residual stresses according theoretical calculations. It is found that the electric field applied during annealing also contributes to the bonding strength. A possible mechanism of influence of the electric field on the bonding process was discussed. The results demonstrate the unique potentials of the bonding technique in fabrication of MEMS devices based on the crystal quartz.

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