Electrostatic Bonding of Silicon-to-ITO coated #7059 Glass using Li-doped Oxide Interlayer

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Silicon to In₂O₃:Sn (ITO) coated glass bonding has been developed for the packaging of Field Emitter Arrays (FEAs) fabricated on silicon wafer. This paper will focus on the processing and results for silicon-to-ITO coated glass bonding using anodic bonding process. Lithium oxide doped layer was deposited on ITO coated glass by electron beam evaporation. The breakdown voltage of lithium doped oxide interlayer was investigated with the comparison of Sputtered #7740. Silicon-to-ITO coated glass bonding occurs in the range of temperatures from 260 °C to 320 °C with applied voltages ranging from 140 V_{DC} to 220 V_{DC} . The bonding strength obtained from tensile test was about 10 MPa under condition of 180 V_{DC} in 320 °C. In order to study the role of the lithium ions in the bonding mechanism, secondary ion mass spectroscopy (SIMS) analysis was carried out. The possibility of the packaging method of FED is proposed.

I. INTRODUCTION

The anodic bonding [1,2] of silicon and glass has become one of the important procedures in the fabrication of microsensors [3]. The anodic bonding is supposed to provide a strong and hermetic seal that protects the silicon chip from the environment. This method has been applied to pressure sensors [4], accelerometers [5], solar cells [6], heat sinks for VLSI [7] and so on. Anodic bonding process was described for the first time by Wallis and Pomerantz in 1969. Since then, Pyrex #7740 glass has been normally used because it has nearly the same thermal expansion coefficient as silicon. Glass to silicon anodic bonding has generally been used for making microsensor and actuator.

Anodic bonding in its basic form is a combined thermal and electrostatic bonding process. Temperature plays an important role in anodic bonding process since it increases the ion mobility in glass [8]. As the glass temperature is raised, the resistivity decreases exponentially and a rapid build up of the space charge occurs at the interface, generating electrostatic field which pulls the surface together. This electrostatic field allows the formation of Si-O-Si bonds originating either from silicon oxidation at the interface or by thermodehydration of silicon-glass ensemble. The formation of a SiO_2 layer between silicon and glass during bonding was proved by backscattering spectroscopy [9].

Nowadays anodic bonding can be applied to FED packaging process by joining Si substrate to glass. FED is currently sealed in a manner in which the two plates are aligned and held together with special fixturing. A solder glass frit material is placed between the plates in the peripheral edge sealing area. Then, the plates are slowly heated to $450\sim600$ °C, depending upon the frit materials. In this method, FEA [10] is fabricated on glass substrate.

Since temperature and voltage are important factor which can affect the anodic bonding process, lowering temperature and applied bias is very important to reduce the thermal stress during bonding process. In this paper, anodic bonding process at condition of low temperature and bias was accomplished.

In this work silicon-to-ITO coated glass bonding has been developed for the packaging of FEA fabricated on silicon wafer. The new packaging method is suggested for the application of the basic sealing procedure in the packaging of FEA fabricated on silicon wafer.

II. EXPERIMENTS

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Fig. 1. SEM photograph of the interface region of ITO coated glass with deposited lithium oxide interlayer.

Lithium oxide doped interlayer was deposited on the ITO coated glass (Corning #7059) substrate by electron beam evaporation. The mixture of SiO_2 and Li_2O was used as the source material of electron beam evaporation. The mixing ration was 1.7 wt% Li₂O of SiO₂ amount. The glass interlayer was deposited at a substrate temperature of 200 °C under the pressure of 3.6×10^{-5} Torr. The glass interlayer was typically 1.5 μ m thick. The glass obtained in this manner was observed by means of cross sectional scanning electron microscopy (SEM). Fig. 1 shows the SEM image of a vertical cross section. In the conventional method, Pyrex #7740 bulk glass or interlayer has been mainly used for the bonding process. So it is impossible to change the metallic ion concentration in oxide layer. However, it enables widening of the range of metallic ion doping concentration in oxide layer by using the SiO_2 and Li_2O mixture.

The samples used for the experiments were 4 inch double-polished silicon wafers, (100), n-type. Then, it was diced into silicon chip of $20 \times 20 \times 0.5$ (mm). ITO coated glass substrates of $50 \times 50 \times 10$ (mm) were used. Prior to the anodic bonding process, each sample was immersed in acetone and cleaned using an ultrasonic



Fig. 2. Schematic experimental apparatus for silicon to ITO coated glass substrate bonding.



Fig. 3. Dielectric breakdown histograms; (a) Sputtered #7740 and (b) lithium doped oxide interlayer.

cleaner for 5 minutes. The cleaned specimens were rinsed in D.I. water and ultrasonically cleaned for again 5 minutes. After a final rinse in water, cleaned specimens were dried for 10 minutes at 150 $^{\circ}$ C to remove water on surface.

The apparatus for the bonding process consists of hot plate on which the parts to be bonded are heated to temperatures between 260 °C and 320 °C and a DC-power supply which is connected to the two parts in such a way that is negatively charged with respect to glass (Fig. 2). Anodic bonding was performed at the temperature of 260 °C~320 °C with an electrostatic of 140~220 V_{DC}. The voltage was applied for duration long enough to allow the current to settle.

III. RESULTS AND DISCUSSION

In order to investigate the characteristic of breakdown voltage, Al dots having 3000 Å thickness, and 0.07 cm in diameter were deposited on the lithium doped oxide layer by thermal evaporator. The distribution of breakdown



Fig. 4. SEM photograph of the bonded interface; (a) Silicon to ITO coated glass assembly and (b) Magnification of bonded interface.

fields was measured by the time-zero dielectric breakdown (TZDB) technique. Fig. 3(a) shows the breakdown voltage of #7740 interlayer by sputtering method. The average breakdown voltage was 2.1 MV/cm. However, lithium doped oxide interlayer by e-beam has a relative higher breakdown voltage value than that for sputtering method as shown in Fig. 3(b). The average breakdown voltage was 3.2 V/cm. In the anodic bonding process, interlayer breakdown occurred frequently due to low breakdown field characteristics. As this phenomenon occur at lower voltages than those necessary to bonding, it will induce non-bonded region in specimen. Sputtered #7740 has been mainly applied to anodic bonding. Since the breakdown voltage of e-beam evaporated lithium oxide interlayer was higher than that of sputtered #7740, which were mainly used in the conventional method, it is thought that this technique will be more widely used than the conventional one.

The bonded specimen was cut with a diamond cutter and polished in order to inspect the bonded interface region of the silicon to ITO coated glass substrate assem-



Fig. 5. Tensile strength of bonding. (a) different voltage and (b) different temperature.

bly. Fig. 4(a) shows the cross sectional SEM of silicon to ITO coated bonding interface and Fig. 4(b) shows a magnified image of the silicon to glass bonded interface. ITO layer was corresponding to the bright one in Fig. 4(a). Also, glass substrate (Corning #7059) underneath the ITO layer and lithium oxide doped interlayer (dark black) above the ITO layer were shown. The bonding interface was found between the lithium oxide doped interlayer and silicon (bright black). Hermetic sealing is an important aspect of the fabrication of many micromechanical applications. Even an extremely small gas



Fig. 6. Optical photographs of fracturing bulk glass after tensile test.

leakage along bonding interface will lead to a significant pressure increase inside the sealed cavity. From these figures, it was confirmed that there is no micro-gap which can give rise to gas leakage. Therefore, it is thought that it enables this technique to apply the vacuum device.

The mechanical strength of the bonding was measured by using a tensile test. The chips were loaded gradually until the bonded interface broke apart. The bonding strength depends on the surface roughness and particles. In addition, bonding strength has a relation to temperature and applied bias.

Fig. 5(a) shows the measured bonding strength for the applied voltage during bonding process and Fig. 5(b) for the bonding temperature. As can be seen, the bonding strength generally increases with the applied voltage and temperature. The maximum bonding strength was about 10 MPa under the applied voltage of 180 V_{DC} and temperature of 320 °C. Though it was attempted to measure the bond strength above 220 V_{DC} and at 320 ^oC, it was impossible owing to fracturing bulk silicon or bulk glass. Fig. 6 shows the optical image of fracturing phenomenon in bulk silicon after tensile test. From this result, we know that the bonding strength of silicon to glass bonding using lithium doped oxide interlayer has a higher value than that of bulk silicon. This result indicates that it enables this technology to apply the vacuum device as an ideal hermetic seal package is realized.

In order to study the role of the lithium ions in bonding mechanism, secondary ion mass spectroscopic (SIMS) analysis was carried out. Fig. 7 was obtained by SIMS that used cesium ions(Cs^+) incident on the deposited lithium oxide doped layer to cause negative secondary ion to be ejected. From the spectra, other atoms except lithium ion did not show any significant differences between pre-bonding and post-bonding. The lithium ions



Fig. 7. Depth profile of the deposited lithium oxide doped interlayer; (a) Depth profile of pre-bonding and (b) Depth profile of post-bonding.



Fig. 8. Vacuum packaging of FED fabricated on silicon wafer using silicon-to-ITO coated glass bonding.

were uniform in the lithium oxide doped layer of prebonding, as shown in Fig. 7(a), whereas they were almost depleted from the surface region of the deposited lithium oxide doped interlayer of post-bonding, as shown in Fig. 7(b).

In order to provide a strong and hermetic seal, it is necessary for silicon and glass to have a very intimate physical contact. For anodic bonding, this contact can be achieved by applying the DC-voltage across silicon and glass. Without DC-voltage, silicon and glass are separated by a gap of several μ m. The applied voltage generates an electrostatic force that pulls the parts into close -S410-

contact. When this electrostatic force is formed, Li^+ ions that are mobile at the bonding temperature move along the electrostatic field from the glass/silicon interface to the negative electrode where they are neutralized. This causes a depletion of free positive ions (Li^+) in the region near the glass/silicon interface. The remaining fixed negative ions (O^{2-}) create a space charged region. These fixed negative ions react with silicon, leading to the formation of Si-O bond.

The packaging method of FED is shown in Fig. 8. In the conventional method, FEA was fabricated on glass substrate. But in this study, the previously method is suggested as a new packaging process of FEA fabricated on silicon wafer.

IV. CONCLUSIONS

Silicon to ITO coated glass bonding with deposited lithium oxide doped interlayer was performed using anodic bonding. The anodic bonding is performed at 260 °C~ 320 °C and 140 V_{DC} ~220 V_{DC} . The bonding strength was measured approximately 10 MPa under the condition of 180 V_{DC} at 320 °C. The bonding strength generally increases with applied voltage and temperature. From the SIMS results, the role of lithium ions in the silicon to ITO coated glass bonding mechanism is proposed. Silicon to glass anodic bonding is favorable as a packaging method for Field Emission Display.

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