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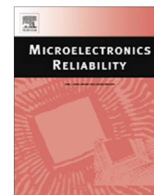

# Tensile tests of micro anchors anodically bonded between Pyrex glass and aluminum thin film coated on silicon wafer

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ABSTRACT

Micro anchor is a kind of typical structures in micro/nano electromechanical systems (MEMS/NEMS), and it can be made by anodic bonding process, with thin films of metal or alloy as an intermediate layer. At the relative low temperature and voltage, specimens with actually sized micro anchor structures were anodically bonded using Pyrex 7740 glass and patterned crystalline silicon chips coated with aluminum thin film with a thickness comprised between 50 nm and 230 nm. To evaluate the bonding quality, tensile pulling tests have been finished with newly designed flexible fixtures for these specimens. The experimental results exhibit that the bonding tensile strength increases with the bonding temperature and voltage, but it decreases with the increase of the thickness of Al intermediate layer. This kind of thickness effect of the intermediate layer was not mentioned in the literature on anodic bonding.

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## 1. Introduction

Micro anchor is a kind of typical structures in Micro-Electro-Mechanical Systems (MEMS) devices, which are made of silicon wafers utilizing surface and bulk micromachining processes [1]. Usually, the anchor structure plays an important role in MEMS sensors (e.g. MEMS inertial micro accelerometers and gyroscopes) and acts as a mechanical or electrical connecter between the movable structures and the static substrate. As a common micromachining process in MEMS industry, anodic bonding technique is utilized to construct the micro anchors. Initially, anodic bonding is developed to bond silicon (Si) wafers with and without oxide films [2], mostly for the fabrication of silicon-on-insulator (SOI) substrates [3,4], but at present, it has matured to a flexible technology with applications far beyond its original area of SOI substrates [5]. Anodic bonding, also known as field-assisted bonding or electrostatic bonding [6], was originally developed in the late 1960s by Wallis and Pomerantz as an effective method to bond metals, alloys or semiconductors to conductive glasses, such as Pyrex glass. This process has become one of the most important silicon packaging techniques in the semiconductor device industry, especially in the MEMS field. It was reported that anodic bonding became a highly promising method for joining certain metals, such as Al, or semiconductors to alkali–ion–conductive glasses at a relatively low temperature [7]. Compared to other wafer bonding techniques, the significant advantages of anodic bonding are as follows: (1) by applying an electric field a strong bond could be acquired at a much lower bonding temperature than the softening point of glass or the melting points of the materials involved [8]; (2) anodic bonding can tolerate rougher surfaces and does not require an ultraclean environment. However, the main disadvantage of anodic bonding for the microelectronic device and MEMS industry is the need to join silicon to a material which is sufficiently electrically conductive at the temperature used for joining [9]. Currently, anodic bonding is used for making relatively simple devices [9], but there exists a demand for this straightforward and reliable bonding technique, in connecting, packaging, or hermetic sealing of more complex micro/nano structures and integrated microcircuits in MEMS devices.

Anodic bonding is a highly coupled physical and electrochemical process. In this complex process, two almost perfectly planar and clean surfaces of two materials must be brought into close contact first. When the bonding temperature is reached, a direct current (DC) voltage is applied for a certain time. The conductive glass is on the cathode side, and the metal, alloy, or semiconductor on the anode side. Once a DC voltage is applied, the mobile cations in the glass, for instance, the sodium cations, move away from the bonding interface to the cathode, a cation-depleted layer is created adjacent to this interface. Intimate contact of the bonding surfaces is achieved by an electrostatic force in the electrical field due to the movement of the cations [10]. A permanent bond is formed by anodic oxidation of the anode material at the interface. The oxygen anions for the oxidation are presumed to originate from either the non-bridging oxygen (NBO) ions in the glass network or the dissolved water in the glass surface [11–14]. When the anodic bonding completed, a special intermediate layer will be formed across the bonded interface and this layer no longer has the same

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material properties as the bonding pairs. Structural reliability of micro anchors will be limited largely by the material properties of that unique intermediate layer. The drop tests [15] of MEMS accelerometers demonstrated that one of main failure modes was the stripping of the micro anchors from their substrates.

There are many approaches to characterize the material properties of the bonding interlayer. The most popular technique is the tensile test [16–18]. There are nearly as many tensile measurement set-ups as authors in this field, which make comparison between reported results difficult [19]. To the author's knowledge, most of the tensile specimens mentioned in forepassed literature were diced from the large bonded wafers. The dicing process inevitably brings about micro cracks at the periphery of the test samples. In addition, the bonding areas in those specimens were much larger than those in practical MEMS devices, which also involved the scale effect of the periphery of bonding area into the experimental results. A device designer usually could not directly take more advantages of those tensile experiments because comparison of tensile strength measurements can be made only between identical test geometries.

Recently, we studied the micro anchors anodically bonded between Pyrex 7740 glass wafer and aluminum thin film coated on crystalline Si wafer [20]. In this article, we develop and report the tensile experiment for anodically bonded micro anchors with actual size and same configuration in actual microaccelerometers. Our objective in this study is to provide some useful experimental results for practical MEMS devices.

## 2. Experimental

### 2.1. Specimen preparation

The tensile specimens, shown in Fig. 1, were made by anodic bonding, which performed at different temperatures and voltages. Pyrex 7740 glass wafers were of 100 mm in diameter with thicknesses of 500 and 700 μm. Their chemical composition includes 80.8 Mol% $SiO_2$, 12.0 Mol% $B_2O_3$, 4.2 Mol% $Na_2O$, 2.0 Mol% $Al_2O_3$, 0.6 Mol% $K_2O$, 0.2 Mol% MgO, and 0.2 Mol% CaO [21]. Si wafers of 100 mm in diameter (double-side polished; *p*-type; wafer surface plane, (1 0 0)), were patterned with 200 μm × 200 μm mesas. These mesa structures were used to avoid the micro edge cracks due to the chip dicing process. The mesas of 2 mm center-to-center spacing and 10 μm in height were prepared with a deep reactive ion etch (DRIE) process. Each 4 mm × 4 mm die contained 4 mesas. The patterned Si wafers were coated by a sputtering system (ARC-12M) with pure aluminum (99.999%) at thicknesses of 500, 950, 1300, 1500, and 2300 Å, respectively. Then these Si wafers were also diced into squares of 12 mm × 12 mm. All square samples were cleaned by deionized water spray rinse in a 100-class clean room and dried by nitrogen gas under pressure. A pair of glass and well-coated crystalline silicon chip was placed between two stainless steel plates and then was set between two hotplates, which acted as plate electrodes. The glass was connected with the cathode side.

The bonding temperature was varied between 300 °C and 375 °C with a bonding voltage between 150 V and 450 V. The DC voltage applied time (bonding time) was 30 min on an open (non-vacuum) bonder, and the bonding voltage was applied when the bonding temperature reached the set point. When the bonding was completed, the bonded sample was cooled to room temperature of 20 °C in 2 h.

### 2.2. Tensile experiment

For the purpose of characterizing the bonding strength, each bonded chip was diced into 4 mm × 4 mm specimens by an automated dicing saw. Before the tensile test, all samples were examined under an optical microscope with 50× objective lens in order to exclude defective ones. The specimens were attached to aluminum jigs with glue for the tensile pulling test. The bonding strength was measured by a tensile pulling machine (GATAN Microtest 2000) and the specimen under test was pulled perpendicular to the bonding interface until its ruptures. The test pulling speed was 1 mm/min, and a personal computer was used to control the pulling speed and the data acquisition. In our preliminary pulling tests, we found the maximum failure loads of these specimens were less than 5 N, and the specimens were easily damaged when they were fixed to the test machine with rigid fixtures. The alignment of the test studs is of critical importance because small deviations will induce bending moments which can cause premature failure of the sample under test [16]. In order to improve the alignment of test samples and to avoid the early failure caused by the force during sample installation, we have designed a fixture with flexible steel wire strand, as shown in Fig. 2.

## 3. Results and discussion

The mean bonding tensile strength are shown in Figs. 3–5, which are averaged from the results of five or more specimens. The measured bonding strength values exhibit a rather large variance around a mean value. This can be attributed to several aspects, such as the misalignment of specimens, the characteristics of bonding behavior [18], the micro defects due to residual stress, and etc. All the specimens pulled apart were also examined with the optical microscope and scanning electron microscope (SEM),

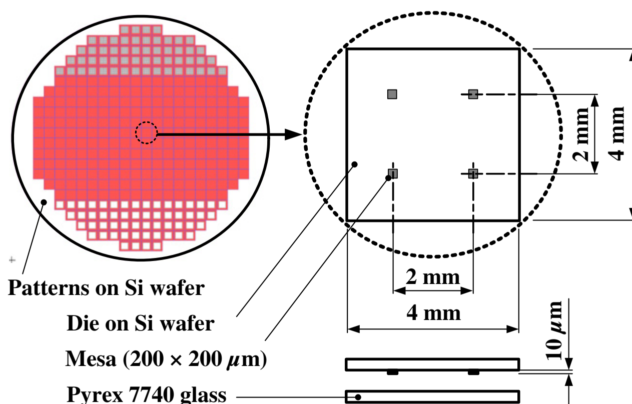

**Fig. 1.** Schematic of patterned silicon wafer. The mesa structures are used to avoid the edge micro cracks due to the chip dicing process.

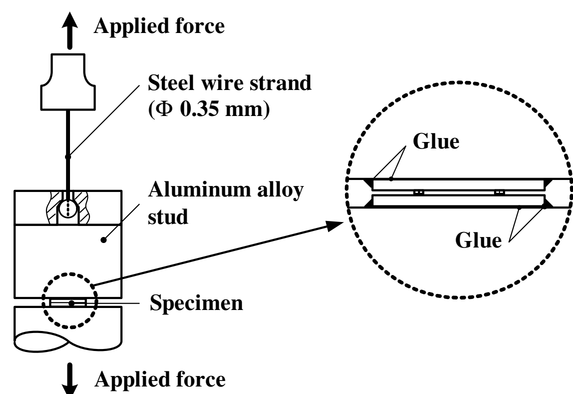

**Fig. 2.** Schematic of fixture for test specimens.

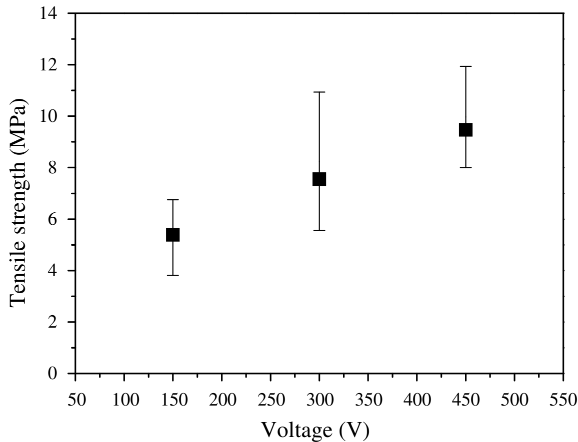


**Fig. 3.** Bonding tensile strength as a function of bonding voltage. Bonding time is 30 min, and bonding temperature is 300 °C. The bonding voltage varies from 150 to 450 V. The specimens contain 700 μm thick Pyrex 7740 glass and 1300 Å thick aluminum film.

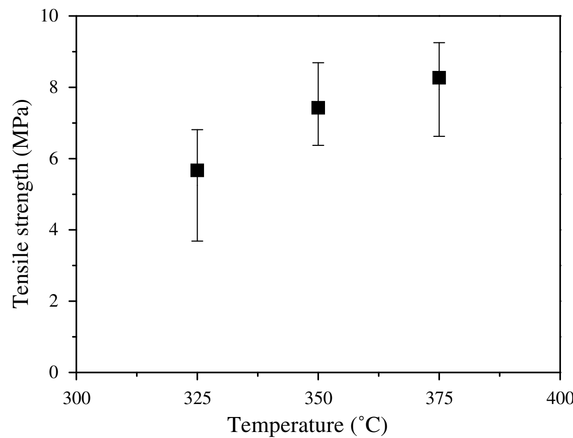


**Fig. 4.** Bonding tensile strength as a function of temperature. Bonding time is 30 min and bonding voltage is 400 V. The bonding temperature varies from 325 to 375 °C. The specimens contain 500 μm thick Pyrex 7740 glass and 500 Å thick aluminum film.

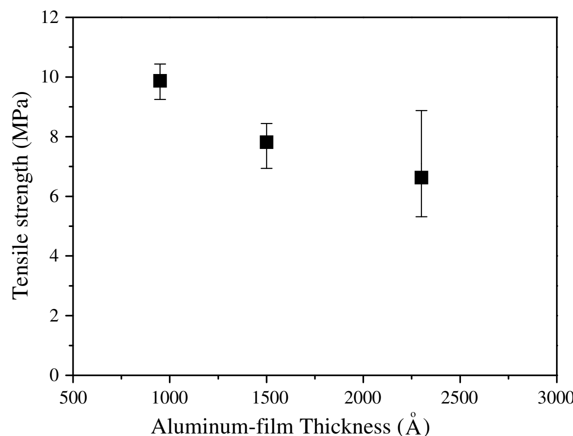


**Fig. 5.** Bonding tensile strength as a function of aluminum film thickness. Bonding temperature is 300 °C, bonding voltage is 400 V, and bonding time is 30 min. The Al film thickness varies from 950 to 2300 Å. The Pyrex 7740 glass used in the specimens is 500 μm thick.

and some parts of the aluminum film and the Pyrex glass were residual on the opposite side, as shown in Fig. 6.

Fig. 3 shows the tensile strength increases with the bonding voltage ranged from 150 to 450 V. The specimens in Fig. 3 were bonded with 700 μm thick Pyrex 7740 glass wafer at 300 °C, and their aluminum film is 1300 Å thick. In the process of anodic bonding, the higher bonding voltage means the larger force produced by the special electrostatic field across the bonding interface, and subsequently brings about much larger and more intimate contact area between the bonding pairs, that is, more chemical bonds generated at the bonding interface and an enhancement of the bonding strength. On the other hand, the higher the bonding voltage is applied, the more amounts of movable ions will be produced in the Pyrex glass and then the more anions, mainly $O^{2-}$, will accumulate in the bonding glass adjacent to the interface of glass/Al, which provides more opportunities to form the chemical bonds between oxygen and aluminum. Unfortunately, however, a high bonding voltage will introduce a risk of electric breakdown, and the maximum bonding voltage is limited mainly by the breakdown voltage over the depleted layer.

The tensile test results tell that the maximum failure loads also increases with the bonding temperature from 325 to 375 °C, as shown in Fig. 4. The specimens used in Fig. 4 were bonded with 500 μm thick Pyrex 7740 glass wafer, and their aluminum film is 500 Å thick. With the bonding voltage of 400 V, the average value of bonding tensile strength goes up from 5.6 to 8.3 MPa. Pyrex 7740 glass has a complicated chemical composition and contains some important alkali elements, which are responsible for the

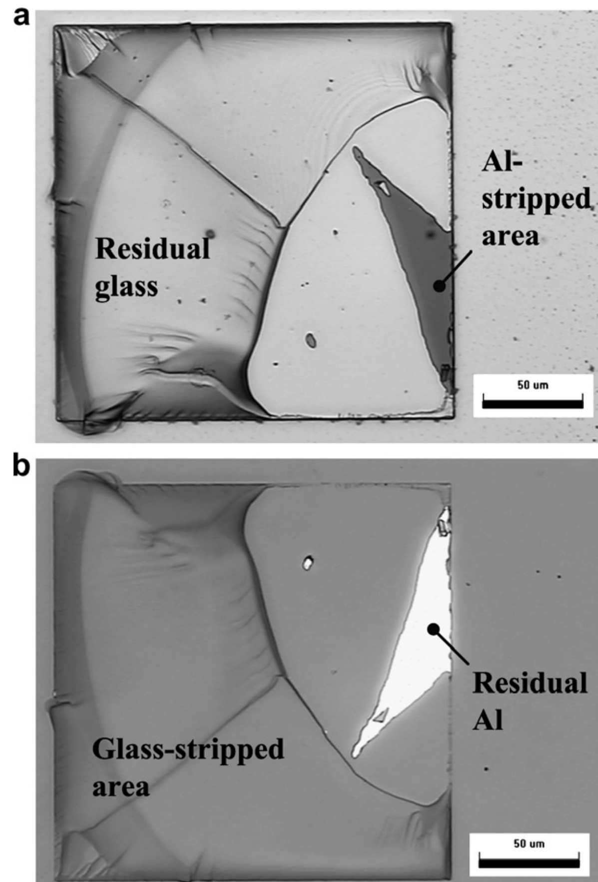


**Fig. 6.** The typical fracture morphology of the samples pulled apart. (a) Silicon-side coated with Al film. Most of bonding area is covered by the residual glass stripped from the Pyrex glass wafer, but some of the aluminum film is stripped off from the silicon substrate. (b) Glass-side. The scale mark in figure is 50 μm.

ionic current in the glass during the anodic bonding process. Due to the low thermal activation energies of alkali ions in Pyrex glass, when the bonding pair is heated, mobile cations, mainly sodium cations, will be easily drift under an electrostatic field. The increasing of bonding temperature gives rise to more amounts of the cations, and correspondingly induces a large number of anions to accumulate near the bonding interface, which also provides more opportunities to produce chemical bonds with aluminum in anode and then improves the bonding strength. But a high bonding temperature not only will lead to the degradation of metal leads and integrated circuits in MEMS device, but also will induce large thermal stress and a high bonding voltage will bring about a risk of electric breakdown. In our high temperature bonding tests, we can find cracks in the glass of some specimens due to thermal stress.

When comparing the tensile failure loads from the specimens which were bonded under the same temperature, voltage, and duration time but with the different thicknesses of aluminum film, we find that their bonding tensile strength increases with the decrease of aluminum film thickness in our tested range, as shown in Fig. 5. All the tested samples in Fig. 5 were anodically bonded under 300 °C, 400 V, and the bonding time of 30 min. The mean tensile strength increases from 6.6 to 9.9 MPa, while the Al film thickness decreases from 2300 to 950 Å, which indicate a fact that the experimental results show the remarkable size effect. It is a clear fact that the bonding strength is affected by the thickness of Al film within the tested range, that is, the bonding strength increases with the decrease of the thickness of Al film within the tested thickness. The studies in literature of wafer bonding seldom concern with such scale effect from the thickness of the intermediate layer. Müller and Stoffel [18] once mentioned that a variation of the oxide thickness in the thickness regime between 60 nm and 500 nm did not affect the bonding tensile strength in the study on tensile strength of low-temperature fusion-bonded silicon wafers.

Typical fracture morphology of the tested samples is shown in Fig. 6. It is obvious that a high quality bonding takes place at either the area where the glass is stripped from Pyrex glass side or the area where the aluminum film is stripped from silicon substrate, and a low bonding arises at the area where the aluminum film is stripped from the interface of Pyrex glass/Al. These tensile results demonstrate that the bonding strength of glass/Al interface is higher than that of glass and aluminum, or the strength of Al/Si interface, at the area where glass is residual on aluminum film or where aluminum film is residual on glass.

## 4. Summary

In summary, at the relatively low temperature and voltage, the tensile specimens were anodically bonded using Pyrex 7740 glass and patterned crystalline silicon chips coated with Al film. To investigate the bonding strength of these specimens with actually sized micro anchor structures, the tensile experiments have been completed with the newly designed flexible fixtures.

In the range of parameters selected in our anodic bonding process, the bonding tensile strength increases with the bonding temperature and voltage. This is in agreement with earlier results reported in the literature. But the experiments exhibit that the tensile strength decreases with the increase of the thickness of Al intermediate layer. Although the measured bonding strength values exhibit a rather large variance around a mean value, the experimental results can be referenced qualitatively. With the dimensions of MEMS devices downward, the scale effect in anodically bonded micro anchors should be considerable during the process of device design. Further investigation is necessary to improve the preliminary work in this study.

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