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Long-term evaluation of hermetically glass frit sealed silicon to Pyrex wafers with feedthroughs

Douglas Sparks, Sonbol Massoud-Ansari and Nader Najafi

Integrated Sensing Systems Inc., 391 Airport Industrial Dr, Ypsilanti, MI 48198, USA

E-mail: dsparks@mems-issys.com

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Abstract
The reliability of bonding silicon to Pyrex wafers using a reflowed glass frit seal is examined in this paper. The Pyrex wafers have metal feedthroughs, which are used to actuate and capacitively sense a single-crystal, silicon resonator. Long-term, high-temperature storage conditions for chips with and without getters are examined. The reflowed glass seal is demonstrated to be hermetic for years at high temperature using both diaphragm deflection and the *Q* and frequency of resonators as a vacuum indicator. The use of a thin film getter is found to eliminate *Q* hysteresis due to gas desorption and adsorption, observed in other resonators studies.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Many microelectromechanical system (MEMS) devices, such as resonators, displays, digital micromirror and microfluidic devices, gyroscopes and tunneling sensors, rely on hermetic, vacuum packaging for improved performance [1–6]. Glass frit, solder, fusion, anodic and eutectic sealing have been used for many years to provide a vacuum seal [1–3, 7–15]. Reflowed glass frit sealing has traditionally been used in pressure sensors, accelerometers and switches to join two silicon wafers together [1, 3, 7–11] with a dielectric seal that can conformally cover minor surface steps. Glass sealing has recently been employed to bond silicon to Pyrex to vacuum seal resonant density and Coriolis mass flow sensors [3]. This paper covers this development and long-term, accelerated life tests that have been undertaken to evaluate the reliability of this sealing method between a silicon wafer and a metallized Pyrex wafer. This process enables chip-level vacuum packaging of MEMS devices as shown in figure 1.

In addition to testing a reflowed glass seal, this study was also expanded to cover the reflowed vacuum seal combined with two generations of thin film getters, used to reduce the microcavity pressure. Without a getter the microcavity pressure is limited by desorption of surface molecules. This desorption can cause sensor hysteresis and is a potential failure mode in high-performance devices. To overcome the surface desorption limit found with wafer bonding, getters have been employed. Metallic getters have been used for decades dating back to vacuum tubes to obtain lower pressures in hermetic packages [16]. Pure metals and alloys are used in cathode ray tubes, flat panel displays, semiconductor processing equipment and other vacuum equipments to lower the pressure. These metals trap various gases through oxide and hydride formation and by simple surface adsorption. Capture of oxygen, nitrogen and hydrocarbons requires elevated temperatures (200–550 °C), while trapping of hydrogen by the metals occurs at room temperature. Getters were first applied to MEMS devices in the mid-1990s [17–19]. In these early studies, nonevaporable getters (NEGs) either in tablet or strip form were placed in an extra micromachined cavity or adjacent to the chip in a ceramic package. To maximize surface area, the NEG is often fabricated using powder metallurgy techniques in which the sintering of the metal particles is just initiated, leaving gaps between metal beads. A high-temperature activation step in vacuum or hydrogen containing a reducing ambient is required to remove the surface oxide layer that forms on the metal particles during the sintering process. This activation step is accomplished by either annealing the whole package or Joule heating of the NEG strip. One problem encountered with sintered getters is particle generation. When NEG metal strips are employed, they are typically cut into a small segment and hand placed

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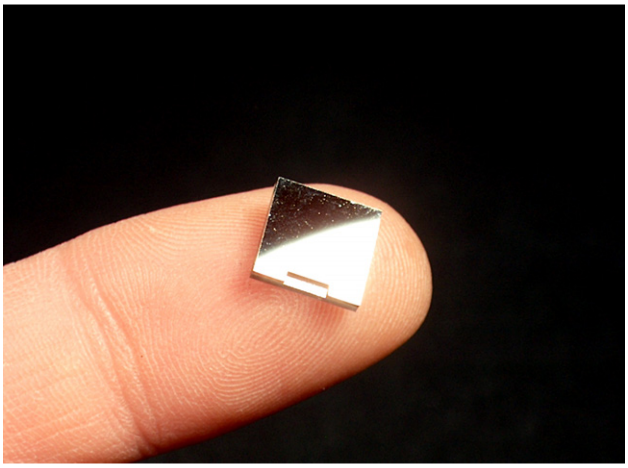

Figure 1. Silicon to Pyrex, reflowed glass sealed MEMS chip.

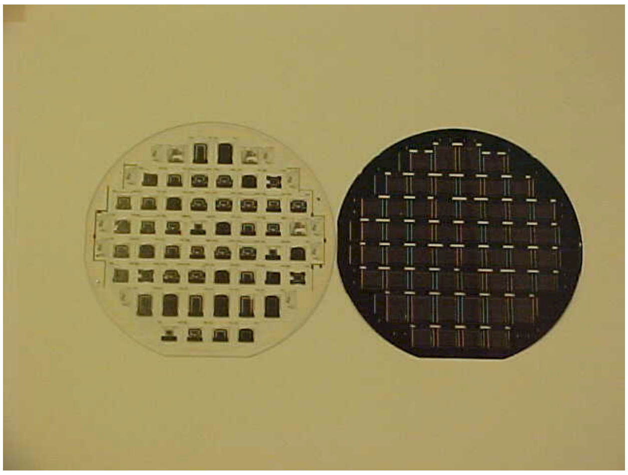

Figure 2. The active metallized Pyrex MEMS resonator wafer, left, and the silicon cap wafer, right, prior to glass frit reflow, wafer bonding.

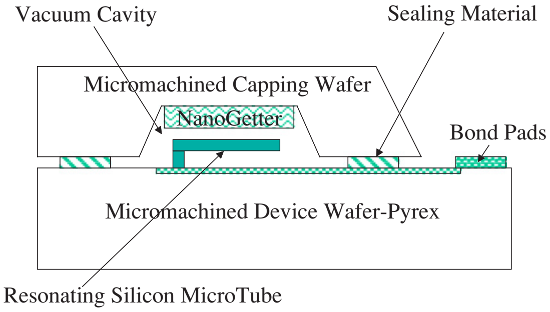

Figure 3. Cross-sectional illustration of the glass sealed vacuum packaging approach using a getter.

into a microcavity prior to wafer bonding. The strips often bend during cutting, requiring additional manual handling to straighten the pieces. Particles are generated during handling and the cutting process. The 2–3 μm diameter metal particles can cause electrical shorts, impede motion and shift resonant frequencies. A frequency shift occurs due to a mass change in the resonator caused by the attached particle. In the last two lifetests of this study multi-metal, thin film getters were employed [20, 21].

2. Experimental proceedings

The micromachined resonators used in this study are solid or hollow silicon resonators [3]. The hollow resonators are used in producing fluidic density sensors or Coriolis mass flow sensors. The resonators are formed by anodically bonding a patterned silicon wafer to a metallized, etched glass wafer. Metal electrodes are formed on the glass wafers to act as drive and capacitive sense elements. The electrodes are linked to the bond pads with narrow feedthroughs. The glass frit, in the form of a thixotropic paste, is screen printed onto the silicon cap wafer and dried. The printed annular glass frit ring will eventually form the hermetic seal for the individual chips. A final wafer bonding process to melt the glass particles is performed next to seal the resonator in a vacuum. The glass reflow and bonding temperature is 400 °C under vacuum. Figure 2 shows the metallized Pyrex wafer with the silicon resonator (left) and the silicon capping wafers with the printed glass seal (right). One of the advantages of the reflowed glass frit sealing process is that the glass can hermetically cover relatively small steps and particles. This is a feature that the anodic, eutectic and fusion bond processes do not provide.

While silicon-to-silicon glass seal integrity can be examined using IR microscopy, Pyrex wafer bonding offers this capability with a common optical microscopy. During the wafer bonding process the glass softens, particles merge and then spread out over the sealing surface. When slowly cooled this glass layer forms a hermetic seal between the two wafer surfaces.

If a thin film getter is to be integrated into the process, the getter is deposited and patterned after the glass seal layer is in place on the silicon capping wafer. The thin film getter is comprised of a proprietary, patent pending, multi-layer structure; the thickness of the thin film layers is in the 5–500 nm range. Since thin film deposition techniques are employed in a cleanroom environment, the thin film getters are virtually particle free compared to an NEG. The thin film deposition method also enhances the ability to easily integrate the getter into a typical MEMS process flow at the wafer level [20]. Adding the thin film getter does not affect the chip size. Vacuum wafer-to-wafer bonding to reflow the glass seal is performed next in this process and the getter traps desorbed gas molecules during the vacuum bonding process without the need for a subsequent getter activation step. A vacuum wafer bonding system, Electronic Visions 501S, was employed in this study. Figure 3 shows a cross-sectional diagram of the final chip-level package, with a getter, after bonding and chip singulation.

An HP 4194A Phase-Gain Analyzer was employed to take test results on vacuum quality, and resonant frequency. The Q was calculated using the frequency bandwidth where the gain for each frequency is 3 dB down from the peak gain. Uncapped reference resonators were tested in a vacuum chamber. A Varian V-250 turbo pump was used to create a vacuum in the test system when examining decapped resonators.

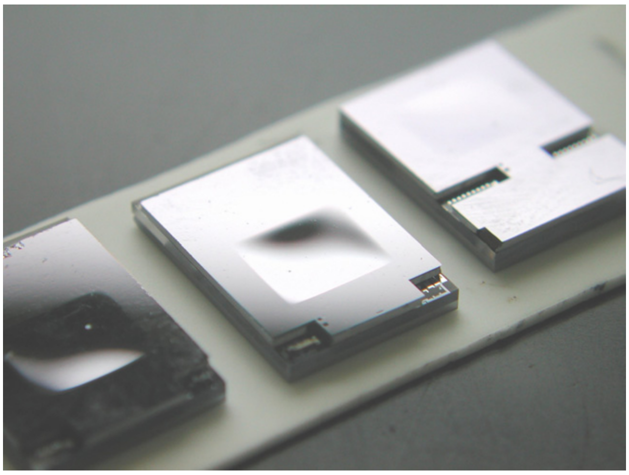

Figure 4. Dimpled, thin diaphragm, denoting a vacuum glass frit seal.

3. Results and discussion

While reflowed glass frit has been used for decades to bond silicon to silicon wafers [7–11], it has not been extensively used to bond silicon to Pyrex since anodic bonding is the traditional method of bonding these two substrates [12]. Since the Pyrex wafer employed in this study to produce the resonant microfluidic devices used metal runners, anodic bonding could not be employed without shorting out the feedthroughs via the silicon cap wafer. To initially test the long-term hermeticity of the basic glass seal, 46 parts were subjected to a prolonged 95 °C bake prior to the development of getter. The higher temperature was selected as a way to accelerate vacuum leaks. These parts were from one complete wafer and several sawn and mounted die, as shown in figure 4. As has been employed by Esashi *et al* [18], the loss of vacuum was monitored during this time by examining the buckling of thinned top-cap diaphragms. As shown in figure 4, when a vacuum is present in the microcavity, a thin diaphragm is dimpled. Pumping these parts down in a vacuum chamber causes the pressure to equilibrate and the downward dimple goes away and can even be reversed. Flat diaphragms were visually observed between 7 and 15 Torr. The diaphragms remained dimpled after several weeks of soaking at 95 °C. Based on these preliminary data, silicon resonators on Pyrex were designed using the glass frit wafer bonding process. The original dimpled parts have been kept in observation over the past three years at 95 °C. Loss of vacuum was only observed when the wafer was accidentally dropped, at which time two diaphragms were cracked. Forty-four of the original parts are still dimpled due to the hermetic vacuum seal after more than three years, or 26 520 h at 95 °C.

As part of the development of the resonant microfluidic devices, a thin film getter was developed and integrated into the process and design to lower the microcavity pressure below 1 mTorr [20]. To evaluate both the hermeticity of the glass frit seal and the absorption properties of the getter, another long-term lifetest was performed. By using the Q value of the vacuum packaged resonators a more quantitative measurement of the cavity pressure could be obtained. To determine what pressure the high Q values corresponded to, a resonator was decapped and tested in a vacuum chamber. Figure 5 shows how the Q varied with pressure for the relatively wide, U-shaped, hollow silicon resonator. With a turbo pump, the chamber pressure could only be pumped down to 790 μTorr (0.10 Pa), which resulted in a Q of 10 350 for the die used to generate the data in figure 5. Plotting $1/Q$ versus pressure results in a linear relationship and an intercept or maximum Q value of 13 065 for this particular device and test conditions. Only a slight increase in Q would be expected at pressures below 790 μTorr. Without the getter, a Q of 40 or a cavity pressure of 1.4 Torr is obtained with reflowed glass frit sealing due to squeeze-film damping and molecular interaction caused by desorbed, trapped gas. The microcavity pressure has been reduced by more than three orders of magnitude through the use of the getter.

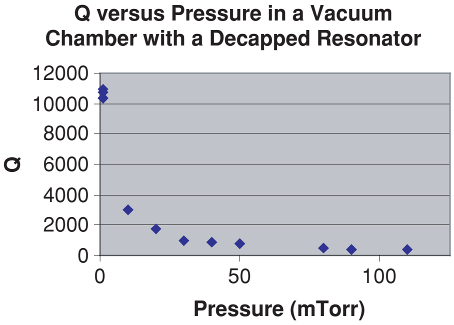

Figure 5. Q versus pressure plot using a vacuum chamber.

Through wafer-to-wafer bonding and the use of the thin film getter, a vacuum level under 790 μTorr, resulting in Q values greater than 60 000 for silicon resonators, can be obtained. Q values have ranged from 2000 to over 60 000 over several years on many separate wafer lots and resonator designs. To further prove the efficacy of thin film getter, in a controlled manner, a silicon cap wafer was partially covered with foil during deposition. At wafer test it was observed that the 18 chips that lacked the getter material had an average Q value of 36 with a standard deviation of 17. The chips with the thin film getter on the same wafer stack had Q values as high as 6760. This controlled experiment with the same wafer proved the effectiveness of the getters in lowering cavity pressure. This getter technology has been applied for several years to chip-scale vacuum packaging of commercially available microfluidic density and chemical concentration meters [3] and micromachined Coriolis mass flow sensors of different flow rate ranges and hence resonator sizes.

The degradation of Q after high-temperature storage from a past study [22] is illustrated in figure 6. These nickel resonators were solder sealed at the wafer level in vacuum without a getter. Since no getter was employed the cavity pressure was in the 1–2 Torr range. At high temperatures, above 50 °C, the Q decreased with time. If the devices were then stored at room temperature, a partial recovery of the Q values was observed. It was postulated that the reversible Q loss and gain were due to gas molecule desorption and adsorption. The nonreversible portion of the Q loss was attributed to either material property changes in the electroformed nickel resonator or ageing in the die attach adhesive.

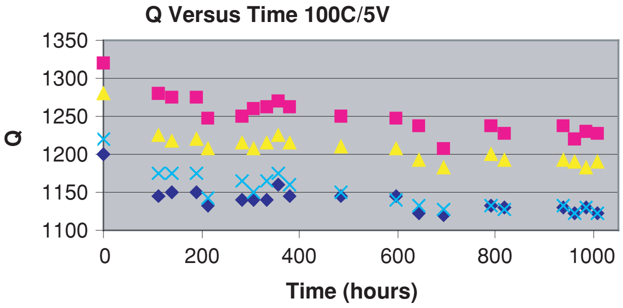

Figure 6. The partially reversible Q loss observed during high-temperature storage of soldered sealed resonant gyroscopes which did not employ getters [22].

Due to the problems seen in Q degradation at high temperature with other resonators, this parameter was studied for the Pyrex mounted silicon devices. In the first long-term test of the getter a variety of different resonators—both hollow and solid—and different designs were employed [21]. The parts, from six different wafers, were periodically removed from the oven, cooled to room temperature and tested using a probe station. Resonators packaged with the getters showed no significant change in Q after storage for 2900 h at 95 °C. Due to the different resonators used in this early test, the standard deviation of Q for both groups ranged from 3000 to 5000. No statistically significant differences between the individual parts and groups were noted during the testing, but the wide group variation made drawing subtle conclusions about Q drift difficult. To investigate potential reversibility of Q due to gas adsorption, both groups of parts were next stored at -10 °C for 240 h. No change in Q was noted after this cold storage step. After this cold storage test, both groups of parts were then stored at 95 °C and monitored periodically. As of 16 100 h, or almost two years at 95 °C, the two groups had average Q values of 5550 and 5271, with standard deviations of 3005 and 4350. Over the course of this extended high-temperature lifetest, the average Q values ranged from 4700 to 8200, with no clear trend. No parts failed to resonate over the course of the study. The parts were mounted to an alumina substrate using epoxy. After several thousand hours at 95 °C, the epoxy went from a light green color to a brown color, indicating ageing. Adhesive ageing could cause a change in clamping losses for a MEMS resonator.

As a follow-up reliability test, a new set of resonators using silicon to Pyrex glass sealing was generated. To minimize both design and packaging sources of Q variation, the same resonator design from the same wafer was used. An improved getter formulation was also employed, resulting in a doubling of the average Q value. Q and frequency values were measured using a probe station at the chip level. No adhesives were used to avoid clamping loss changes due to adhesive ageing effects. Since the probe station was employed, the same electronic amplifier was used with each MEMS chip reducing another source of variation from the study. The parts were soaked at 95 °C, but all measurements were made at room temperature. Figure 7 shows how the Q and frequency varied with time in these parts. The minor difference in frequency can be attributed to variations in room temperature, which would be approximately 3 Hz between the temperatures of 21–25 °C. No significant change in either Q or frequency was observed in the parts. The Q variation is due to the resolution of the Q test method using the 3 dB peak width measurement technique. The average Q value at the start was 23 468 with a standard deviation of 1567. At the end of 3000 h at 95 °C, the average room temperature Q was 24 711 with a standard deviation of 1923. The Q distributions and standard deviations are much tighter in this follow-up study and there is no statistical difference in average Q value after the 3000 h high-temperature soak.

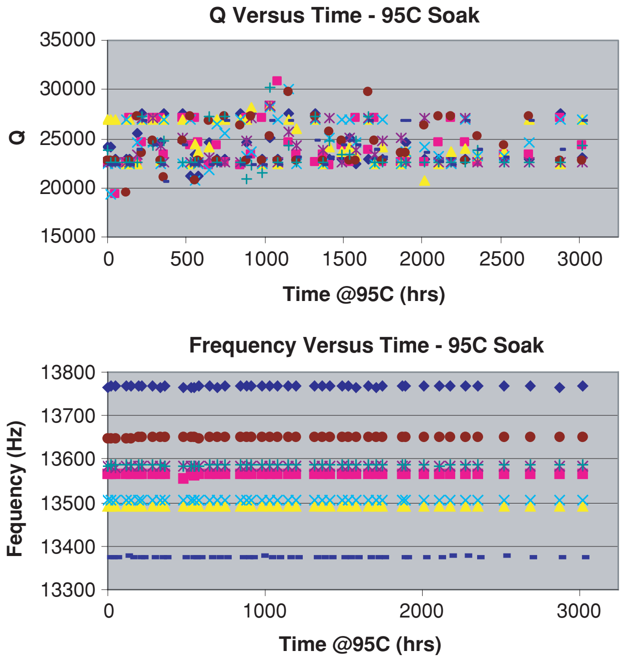

Figure 7. The room temperature Q and frequency variation observed in the third high-temperature storage test.

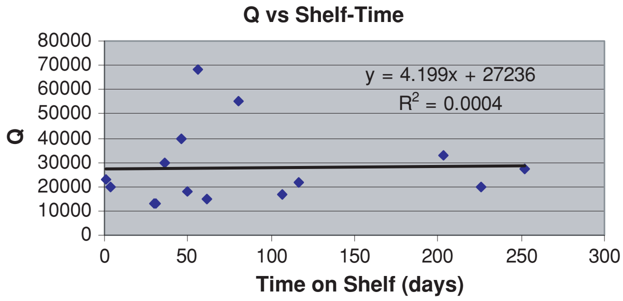

Figure 8. Getter shelf life testing using Q value.

Shelf life and storage conditions prior to packaging of any getter are important considerations. Some getters required vacuum canister packaging or nitrogen cabinet storage prior to activation and use. To gauge the shelf life of the thin film getter formulation used in this study, historical flow card records and wafer-level test results were reviewed for a number of wafers produced over two years. The time between when the thin film getter was applied to the capping wafer and the time that the wafer stack was vacuum wafer bonded together was compared with the average Q value obtained at test. The wafers were stored in a plastic wafer box in a cleanroom, no nitrogen purging was employed during storage, and the wafers were stored in air at room temperature. Figure 8 shows how the Q value varied as a function of getter wafer storage time. No trend was found versus time. High Q values, denoting low cavity pressure and excellent getter operation, were obtained with thin film getters that sat in room temperature air for as long as 252 days prior to wafer bonding.

Other reliability tests have been performed on parts with the reflowed glass seal joining silicon to metallized Pyrex. For commercially available modules employing getter and reflowed glass sealing technology [3] thermal shock from 0 to 125 °C, biased humidity testing at 85 °C, 90%RH, vibration and system level drop testing have also been performed. No loss of hermeticity or getter related particle generation has been observed with this chip-level bonding approach. It should be noted that not only does reflowed glass offer a way of joining metallized Pyrex to silicon with a dielectric seal, it can also be used to join metallized Pyrex to Pyrex to further reduce chip-to-chip coupling of the feedthroughs in high-speed RF and switching applications. This approach enables an entirely insulating, chip-level package to be produced and integrated with metal or silicon micromachined components.

4. Conclusions

The reliability of wafer-to-wafer bonding of silicon to metallized Pyrex was covered in this paper. Long-term (3000–26 520 h) high-temperature storage conditions for chips with and without getters were examined. The reflowed glass seal was demonstrated to be hermetic for years at high temperature using both diaphragm deflection and Q and frequency of resonators as a vacuum indicator. The use of a thin film getter with the glass seal was shown to eliminate Q hysteresis due to gas desorption and adsorption previously observed in resonators studies. When a proper design and process are employed, the reflowed glass bonding approach can produce a reliable vacuum seal with metal feedthroughs.

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