

HYDROGEN DIFFUSION AND PRESSURE CONTROL OF ENCAPSULATED MEMS RESONATORS

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ABSTRACT

We have investigated the diffusion of hydrogen through the oxide and silicon of our single-wafer vacuum package. We have encapsulated micromechanical silicon resonators in vacuum beneath a 20 μm polysilicon layer. While we have not been able to measure any change in pressure of parts at room temperature over a period of nine months, we have been able to accelerate the diffusion of hydrogen through the encapsulation using elevated temperatures. While placing encapsulated resonators in elevated temperature, we select the furnace gas condition to diffuse hydrogen gas in or out. This is an enabling step toward forecasting long-term hermiticity of the encapsulation, and it provides the ability to set the pressure inside the encapsulation with a simple set of furnace processes. Also, the ability of the encapsulation to withstand more than twenty of these temperature cycles between room temperature and 300 $^{\circ}\text{C}$ - 400 $^{\circ}\text{C}$ is evidence of the robustness of the package.

Keywords: Hermetic packaging, Resonator, Hydrogen diffusion, Quality factor

INTRODUCTION

Packaging is frequently the limiting factor in getting MEMS devices into commercial products. There are several necessary traits of a package suitable for MEMS resonators and inertial sensors, including: (1) A known pressure that is stable over time and (2) an environment that is free of constituents that will adsorb and desorb to the device. Work has been done by others to study long term effects [1] and the effects of harsh environmental conditions [2] on MEMS packages. In previous work, we have developed a robust package that can be co-fabricated with the MEMS devices [3]. We have demonstrated that the pressure inside the encapsulation does not measurably change over nine months [4]. To prove this, we use the dependence of resonator quality factor (Q) on pressure. An increase in pressure will lead to a decrease in quality factor for resonators whose Q is limited by gas damping. Therefore, we can monitor the quality factor over time to determine if there has been a change in pressure.

For this work, since the encapsulation has shown no signs of pressure change after nearly a year, we have utilized elevated temperatures to accelerate the hermiticity testing. We have found that, at elevated temperatures of 300–400 $^{\circ}\text{C}$, hydrogen will diffuse in and out of the package on timescales of hours, as opposed to the years necessary at room

temperature. Since this package and resonator can survive repeated exposure to temperatures above 400 $^{\circ}\text{C}$, we can explore the temperature dependence of the hydrogen diffusion and construct a detailed model to support the design of improved packages.

We have studied gas diffusion through our encapsulation for two reasons: (1) to understand high temperature gas diffusion through the encapsulation which will allow us to extrapolate room temperature diffusion constants (2) to investigate the possibility of using high temperature in-diffusion of gas to set the pressure of our encapsulation when damping is desired (*e.g.* accelerometers). As for the particular focus on hydrogen, the only gas species present during the seal of the device are hydrogen, chlorine, silicon, and phosphorous, with hydrogen being the most concentrated gas by a factor of 50. Since hydrogen is the most concentrated species and the most mobile species, it is the focus of this study.

FABRICATION

The end result of our process is MEMS resonators encapsulated in vacuum beneath a 20 μm polysilicon encapsulation. Electrical contact is made *through* the encapsulation layer, such that a finished device appears on the surface to be only metal traces on oxide. The encapsulation layer is deposited, as opposed to bonded, eliminating the need for bond ring area, thereby decreasing the footprint of a finished device [5].

The fabrication proceeds as follows: The resonator structure is etched into an SOI wafer or a wafer with a planarized polysilicon layer on top of a sacrificial oxide layer, fig. 1. The unreleased parts are covered with an LPCVD oxide, and the oxide is etched to allow electrical contact to the resonator structure. A thin polysilicon layer is deposited and defined to allow etching of the sacrificial oxide. The resonator is released with a vapor phase hydrofluoric acid (HF) etch. Using HF in the vapor phase prevents liquid from getting into the resonator structure, preventing stiction, and obviating the need for a critical point dry process. The released devices are sealed in vacuum with a ~ 20 μm polysilicon deposition, and any topology from the underlying structures and deposition roughness is removed with chemical-mechanical polish (CMP). At this point, the resonator is sealed inside the vacuum encapsulation, and only a few steps are necessary to make electrical contact to the resonator. The polysilicon encapsulation layer is etched to electrically isolate the contacts. Oxide is deposited to seal over these etched trenches, and the oxide is etched to allow metal contact to the electrical contacts in the encapsulation

layer. Aluminum is deposited and patterned to finish the devices.

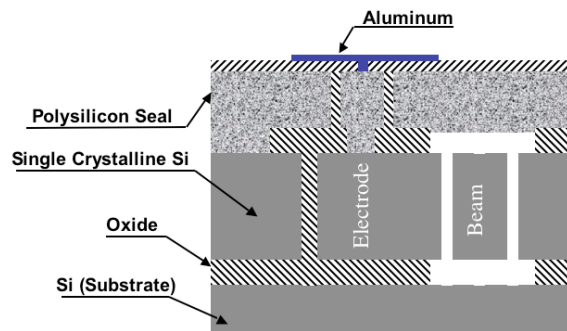


Figure 1. Schematic cross-section of device. Silicon resonator sealed beneath thick (~20 μm) silicon encapsulation layer. Electrical contacts run through encapsulation layer to wafer surface.

An SEM cross section of an encapsulated device is shown in fig. 2.

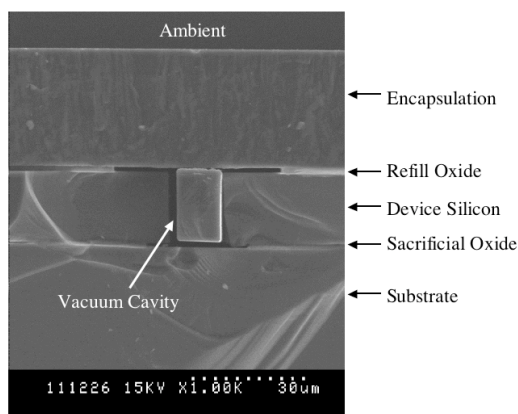


Figure 2. SEM cross-section of device. Vacuum cavity surrounding beam and ambient are labeled.

RESULTS

As stated before, since the quality factor of these resonators is limited by gas damping, any change of pressure inside the cavity results in a change in quality factor. This enables us to use the resonators as pressure sensors for studying pressure changes within the cavity.

Our experiments proceeded as follows: we began with encapsulated resonators ($f_{\text{resonant}} = 297\text{kHz}$) with ~10 mBar of hydrogen in the cavity. We then put them in a furnace with elevated temperature and nitrogen ambient. By putting the encapsulated resonators in a nitrogen-filled furnace at elevated temperature, we can drive hydrogen out of the encapsulation. The high temperature increases the rate of hydrogen diffusion. The nitrogen ambient, free of hydrogen, forces a concentration gradient of hydrogen pressure between the package and the ambient. The nitrogen, which diffuses much more slowly than hydrogen, did not diffuse into the cavity in any measurable amount. We remove the resonators and test the Q for at least seven resonators. Since Q is pressure dependent in resonators that are gas damping

limited, we can detect a change in pressure inside the encapsulation by a change in Q of the resonator. Repeating this furnace treatment and measurement cycle gives a relationship of Q vs. time spent in the nitrogen furnace, fig. 3. Measurement noise, probably from imperfect probe contact, caused some variation in measured Q .

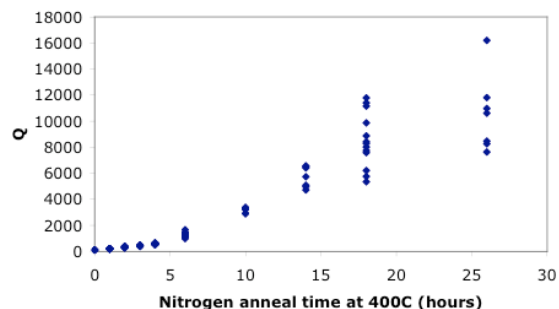


Figure 3. Quality factor increases as encapsulated resonators are in hydrogen-free environment. Increasing quality factor implies decreasing pressure for devices limited by gas damping.

The dependence of Q on pressure for this design of resonator can be experimentally determined by placing a resonator, encapsulation removed, in a vacuum chamber and sweeping the pressure. The relationship of Q vs. pressure for this design of resonator is given in equation 1. This equation is used to convert Q to encapsulation pressure.

$$Q = \frac{1388}{P^{0.98155}} \quad (1)$$

The equation is valid for the region above 0.2 mBar, where the device is primarily limited by gas damping. Below that pressure, other energy loss mechanisms dominate the Q . The experimentally determined Q vs. pressure relationship has similar form to gas damping theory, $Q \propto 1/P$ [6], fig. 4.

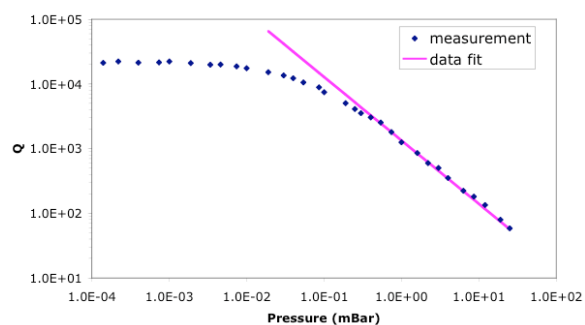


Figure 4. Q vs. pressure for a resonator with encapsulation removed. Above 0.2 mBar, data is pressure limited and well described by $Q = 1388/P^{0.98155}$ which is inline with the $Q \propto 1/P$ relationship expected from simple gas damping theory.

The pressure change inside the encapsulation caused by diffusion will be of the form of equation 2.

$$P(t) = P_{ambient} + (P_0 - P_{ambient})e^{-Dt} \quad (2)$$

P_0 is the initial pressure inside the encapsulation, $P_{ambient}$ is the ambient pressure, D is a coefficient of diffusion with unit (time^{-1}), and t is time. As $t \rightarrow \infty$, $P \rightarrow P_{ambient}$. D is related to the speed at which this take place. For our tests, we assume the partial pressure of the hydrogen inside the nitrogen-filled furnace is vanishingly small, $P_{ambient} \approx 0$, which reduces equation 2 to equation 3.

$$P(t) = P_0 e^{-Dt} \quad (3)$$

After test at 400 °C, the cavities were refilled with hydrogen using a hydrogen filled furnace at 400 °C and the tests were repeated in nitrogen at 300 °C and 350 °C. The decreasing pressure with overall time is shown for the three temperatures, fig. 5.

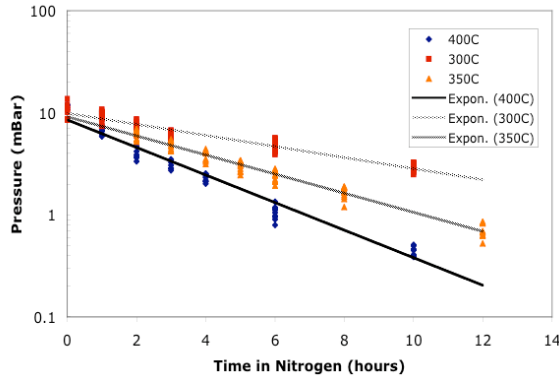


Figure 5. Change of cavity pressure after time in hydrogen-free environment. Pressure inferred from resonator quality factor, which was calibrated in fig. 4. Decreased pressure is caused by out-diffusion of hydrogen from the cavity. Out-diffusion is accelerated by increased temperature. Data fits diffusion equation $P = P_0 e^{-Dt}$ where D is the diffusion coefficient and t is time.

As can be seen, the pressure follows an exponential decay form, where a diffusion coefficient can be extracted for each temperature. Comparing the temperature dependence of the diffusion coefficient gives equation 4.

$$D = 60e^{-\frac{3529}{T}} \quad (4)$$

T is the package temperature in Kelvin. A plot of diffusion coefficient vs. temperature is given, fig. 6.

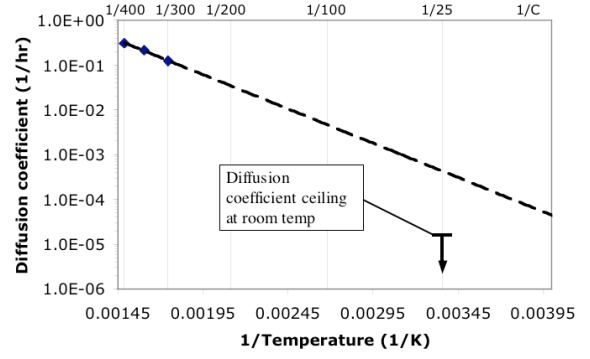


Figure 6. Arrhenius plot of diffusion coefficient vs. temperature. Extrapolation shows that the coefficient is 3 orders of magnitude smaller for room temperature than for 400 °C. The above relationship is described by the equation $D = 60 * e^{-3529/T}$ where D is the diffusion coefficient and T is the temperature. A ceiling for diffusion coefficient at room temperature is found from long-term measurements.

Separate long-term measurements give a maximum diffusion coefficient for 30 °C. The extrapolation from the high temperature measurements exceeds the upper limit from the low-temperature measurements, indicating a complex hydrogen diffusion process in these materials that is not completely described by a simple exponential relationship [7].

It is important to note that the diffusion constant is species specific. Hydrogen, being the smallest element, is likely to have one of the smallest diffusion coefficients, even when it diffuses as H_2 .

While the hydrogen time constant, $\tau = \frac{1}{D}$, may be as small as thousands of hours, there are three mitigating factors that suggest very long term hermeticity of the encapsulation. (1) Long term experimental results previously obtained bound the diffusion time constant to greater than 50,000 hours. The limiting factor from these long term experiments is thought to be from measurement noise, and not from actual diffusion of gas into the encapsulation. The difference between the room temperature time constant extrapolated from high temperature data and the time constant measured at room temperature are, as stated above, an indication of the complex dependence of diffusion on temperature. (2) Other species that might diffuse in will be larger and are likely to diffuse more slowly. A piece of supporting evidence comes from the nitrogen furnace steps that were used to diffuse hydrogen out of the encapsulation. If the nitrogen could diffuse in at a rate comparable to the rate at which hydrogen diffuses out, the Q would not increase over time. If the nitrogen could diffuse at a rate slower than the hydrogen but in the same order of magnitude, the pressure would decrease, then increase. This would signify that hydrogen leaves the encapsulation, and then nitrogen diffuses in gradually. However, neither of these behaviors was observed, indicating that the rate of nitrogen diffusion through the encapsulation is *much slower* than the rate of hydrogen diffusion. This leads to the third factor. (3) There is a very small percentage of hydrogen in the atmosphere. Hydrogen

only makes up 0.00005% of the atmosphere [8], so even if the inside of the encapsulation were equilibrated with the partial pressure of atmospheric hydrogen, the pressure inside the cavity would still be very low. Helium, the next smallest element, is also a low fraction of the atmosphere, 0.0005%. The most abundant element, nitrogen at 78.08%, was determined to diffuse very slowly through the encapsulation, as evidenced by its lack of an effect on encapsulation pressure at high temperature nitrogen furnace processes.

FUTURE WORK

While conclusive results can be made for the diffusion properties of this package, there is more to be learned about the specific paths of diffusion. Specifically, we plan to determine the structure in our encapsulation that is the limiting factor in diffusion (*e.g.* diffusion through the silicon encapsulation, diffusion through the oxide underneath the metal contacts). To do this, we will design structures that selectively enhance or retard diffusion through each of the possible diffusion paths. Also, the intermediate temperature range between 300 °C and room temperature can be further investigated.

CONCLUSIONS

We have utilized a high temperature nitrogen environment to enhance diffusion of hydrogen through a wafer-scale vacuum encapsulation. All tests were performed on *fully functional encapsulated resonators*. The package was able to withstand more than twenty cycles between room temperature and 300 – 400 °C without showing any signs of cracking or failure of the encapsulation, providing strong evidence of the robust nature of this encapsulation process. The increased diffusion rate from the high temperature steps allow for diffusion information to be extracted. In doing so, we have been able to place an upper limit on the diffusion rate at room temperature. The characteristic time constant of diffusion at room temperature was shown to be greater than 1000s of hours, and it is likely closer to 100,000s of hours or more.

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REFERENCES

- [1] J. Chae, J. M. Gianchino, and K. Najafi, "Wafer-Level Vacuum Package with Vertical Feedthroughs," presented at MEMS 05, 2005.
- [2] M. Chiao and L. Lin, "Accelerated hermiticity testing of a glass-silicon package formed by rapid thermal processing aluminum-to-silicon nitride bonding," *Sensors and Actuators A*, vol. 97-98, pp. 405-405, 2002.
- [3] R. N. Candler, W.-T. Park, H. Li, G. Yama, A. Partridge, M. Lutz, and T. W. Kenny, "Single wafer encapsulation of MEMS devices," *IEEE Transactions on Advanced Packaging*, vol. 26, pp. 227-232, 2003.
- [4] B. Kim, R. N. Candler, M. Hopcroft, M. Agarwal, W.-T. Park, J. Li, and T. W. Kenny, "Investigation of MEMS Resonator Characteristics During Long-Term and Wide Temperature Variation Operation," presented at 2004 ASME International Mechanical Engineering Congress and RD&D Expo, 2004.
- [5] W.-T. Park, R. N. Candler, V. Ayanoor-Vitikkate, M. Lutz, A. Partridge, G. Yama, and T. W. Kenny, "Fully Encapsulated Sub-millimeter Accelerometers," 2005.
- [6] W. E. Newell, "Miniaturization of Tuning Forks," *Science*, pp. 1320-1326, 1968.
- [7] N. H. Nickel, "Hydrogen Diffusion Through Silicon/Silicon Dioxide Interfaces," *J. Vac. Sci. Technol. B*, vol. 18, pp. 1770-1772, 2000.
- [8] R. G. Barry and R. J. Chorley, *Atmosphere, Weather and Climate*, 1998.