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Long-term stability of single-crystal silicon microresonators

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Abstract

The long-term stability of single-crystal silicon microresonators is evaluated. The vacuum-encapsulated length-extensional mode resonators (fr ∼ 13 MHz) are demonstrated to be capable of a ppm-level aging rate for a follow-up period of 42 days. Comparison with unsealed devices reveals that the silicon microresonators are sensitive to water contamination from the ambient humidity.

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1. Introduction

A frequency reference is a fundamental requirement for practically every wireless transceiver. The stand-alone reference is needed especially in transportable devices (e.g., mobile phones and radio modems) to quickly establish the communication after power-up. The typical long-term stability requirements span from 10^{-7} accuracy in satellite navigation to 10^{-6} accuracy in mobile phones and 10^{-5} accuracy in close-range wireless systems (Bluetooth, ZigBee) [1,2].

The conventional solution for the frequency reference is a quartz-crystal oscillator [1,3]. The good material stability of quartz allows low aging rates that meet the strict frequency precision requirements [4]. Furthermore, the quartz oscillators can provide excellent spectral purity (low-phase noise) for their output signal, which is utilized in receivers for improved sensitivity and frequency selectivity [5]. The central disadvantage of quartz crystals is their large size which makes the frequency reference a significant space consumer in the modern, otherwise highly integrated transceiver realizations.

Reference oscillators based on micromechanical resonators [6,7] could offer significant savings in circuit board area and manufacturing costs in comparison with quartz crystals. Indeed, our work has shown that via proper design and fabrication, the micro-oscillators can produce phase noise comparable to quartz crystals [8]. However, regarding the long-term stability of the microresonators, there exists very little published work. In this paper we characterize the aging of the length-extensional mode microresonators (fr ∼ 13 MHz) made of single-crystal silicon. For the properly vacuum encapsulated devices our measurements demonstrate a ppm-level long-term stability during a 42-day follow-up period. The results appear promising for obtaining reference-quality long-term stability from microresonators.

2. Sample preparation and measurements

A sample of the studied micromechanical resonators is illustrated in Fig. 1. The resonator vibrates in the length-extensional mode with the first eigenfrequency at fr = 13.1 MHz. The vibration mode is based on the propagation of bulk acoustic wave inside the resonator arms [7,9]. The resonator arm length L = 145 μm corresponds approximately to a quarter of the acoustic wavelength λ/4. The balanced vibration of the two arms leads to a minimal motion at the central anchoring bridge reducing the energy losses to the substrate. The typical recorded quality factor of the resonance is Q = 170 000 in vacuum (p < 1 mbar) and Q = 2000 at normal pressure (p = 1 bar).

The resonator was actuated electrostatically using the narrow gaps (d = 1.0 μm). For improved electromechanical coupling the triangle-shaped end-points of the resonator were used in the design [9]. The resonance of the
The two arms exhibit length-extensional vibration mode illustrated by the black arrows. The end-points of the arms are widened (triangle-shape design) for improved electromechanical coupling. The fundamental resonance frequency is $f_r = 13.1$ MHz and $Q = 170,000$ in vacuum.

The studied devices was detectable even at the normal atmospheric pressure with a reasonable accuracy. This allowed comparison of the vacuum encapsulated devices with unsealed ones operating in normal atmospheric humidity and pressure.

The resonators were fabricated using deep reactive ion etching (DRIE) of silicon-on-insulator wafers (10-μm thick device-layer) [7,9]. We emphasize that the resonators were thus made of single-crystal silicon instead of polycrystalline silicon often used for microresonator fabrication [6]. The release of the moving structures were performed in HF wet etch [7]. Super-critical CO$_2$ drying was used to prevent sticking.

To encapsulate the components, the chips were glued and wire-bonded to standard ceramic dual-in-line (DIL) packages (Fig. 2). The packages were placed in a vacuum chamber that was pumped to a pressure $p = 0.01$ mbar and heated to a temperature of $T = 400$ °C. This procedure was aimed to remove water from the surfaces inside the vacuum chamber, in particular from the resonator and the package surfaces to become encapsulated (Fig. 2a). After a relatively short drying period of 15 min at elevated temperature, a brass cap was lowered into contact with the DIL-package to form the solder joint.

The sealing joint was formed using Sn(62%)-Pb(38%) solder material. Subsequently the system was cooled down in 30 min. Two of the samples (Samples 1 and 2) were encapsulated in the way described above and another two samples (Samples 3 and 4) were left unencapsulated for comparison purposes.

The resonance data for Samples 1 and 2 was recorded before the encapsulation in a laboratory vacuum chamber at different pressures ($p$ ranging from 0.01–1000 mbar). The $Q$-value recorded from the sealed samples was $Q \sim 90,000$. Comparison with the pre-encapsulation data gave an estimate of $p \sim 5$ mbar for the residual pressure inside the sealed packages (Fig. 3). No change in the vacuum level in the sealed packages could be detected during the 42-days follow-up period.

The measurement system was based on a HP 4396B network analyzer in transmission mode. The DIL-packages were attached to a multi-channel measurement system that allowed a simultaneous monitoring of all the samples. The resonators were biased using an external bias voltage of $U_{bias} = 40$ V and the excitation was done with $U_{ac} = 0.142 \times V_{rms}$. The bias voltage was turned on constantly. The transmission response of the resonators was recorded at 15-min intervals. For measurement, the samples were installed into a steel container that was successively

![Fig. 1. Scanning electron microscope view of the studied microresonator.](image1)

![Fig. 2. The studied samples were encapsulated using standard DIL-packages.](image2)

![Fig. 3. The recorded resonance data before encapsulation in laboratory vacuum ($p = 0.01$, 10 mbar) and after encapsulation. The calculated motional resistances at $U_{bias} = 40$ V are $R_m = 364$, 751, 526 kΩ, respectively.](image3)
placed inside a climate chamber. The temperature inside the chamber was measured using a Pt100 sensor which was placed in good thermal contact with the DIL-packages. The temperature information was needed in order to decompose the temperature-induced variations from the recorded frequency data. The climate chamber was typically set to hold a constant temperature \( T = 30 \degree C \). The climate chamber was also used to subject the resonators to temperature cycling in order to determine the resonance frequency temperature coefficients and to test for possible temperature hysteresis effects. A humidity measurement capability was included into the measurement set-up.

3. Results

The recorded change in resonance frequency \( df/dT \) of the resonator Samples 1 and 2 is presented in Fig. 4.

![Fig. 4. The change of resonance frequency for the two encapsulated samples. The temperature-induced variations are removed from the data based on the recorded Pt100-temperature (shown on the right-axis) and the measured temperature coefficient \( 1/|df/dT| \sim -28 \text{ppm/K} \).](image)

The change of resonance frequency \( df \) of the four measured samples. Samples 1 and 2 are encapsulated and Samples 3 and 4 are left open. The temperature-induced variations are removed from the data based on the recorded Pt100-temperature and the measured temperature coefficient \( 1/|df/dT| \sim -28 \text{ppm/K} \). The right-axis shows the measured ambient relative humidity.

![Fig. 5. The change of resonance frequency \( df \) of the four measured samples. Samples 1 and 2 are encapsulated and Samples 3 and 4 are left open. The temperature-induced variations are removed from the data based on the recorded Pt100-temperature and the measured temperature coefficient \( 1/|df/dT| \sim -28 \text{ppm/K} \). The right-axis shows the measured ambient relative humidity.](image)
During the follow-up time of 1000 h (42 days), the encapsulated Samples 1 and 2 have maintained their initial frequency extremely well. Sample 2 shows a minor ∼1 ppm frequency drift. To analyze the temperature hysteresis on the resonators, two elevated temperature cycles were performed as revealed by the temperature data in Fig. 4. During the cycles, the temperature of the climate chamber was raised to $T = 60^\circ C$ for 8 h in the first cycle and for 4 h in the second cycle. No hysteresis (deviation from the original frequency as $T = 30^\circ C$ was restored) was noticed for Sample 1, while for Sample 2 two minor (approximately $-1$ and $-0.5$ ppm) frequency jumps coinciding with the two temperature cycles are observable. The gap in the data in Fig. 4 (at $t = [650, 850] h$) corresponds to the resonators being completely removed from the measurement set-up for a period of nine days. It is evident that the removal induced no drift in the frequencies of the two samples.

Fig. 5 illustrates the dramatic improvement to the stability due to vacuum encapsulation. The non-encapsulated Samples 3 and 4 (prepared and measured simultaneously with Samples 1 and 2) show a rapid initial drift during the early stages of the measurement and a continuous drift thereafter. The magnitude of the drift is several tens of ppm. The measured relative humidity (RH) in Fig. 5 shows a clear correlation with the frequency data from Samples 3 and 4. The calculated correlation factors are $r = 0.88$ and $0.85$ for Samples 3 and 4, respectively. This suggests that the resonator surface is very sensitive to surrounding gaseous water as expected based on existing literature [10]. The increased humidity leads to increase in water absorption of the surface and consequent decrease in resonance frequency. This emphasizes the importance of proper water removal during the vacuum encapsulation procedure for the resonator stability.

4. Conclusions

Using vacuum encapsulated samples we have demonstrated that the studied micromechanical single-crystal resonators ($f_r = 13.1$ MHz) are capable of ppm-level aging behavior during a follow-up period of 42 days. The comparison with unsealed samples has revealed that the resonator stability can be severely affected by water contamination of the resonator surface due to the humidity in the ambient air. The water contamination should thus be carefully taken into account using a dry vacuum sealing procedure, as expected also from the hermetic sealing being a standard method employed for quartz crystals.

We emphasize three aspects. (i) The measured resonators were made of single-crystal silicon instead of polycrystalline silicon often used for microelectronics. The stability obtained using the polycrystalline resonators is under investigation. (ii) The studied devices utilized the length-extensional vibration mode. Such resonators exhibit a much smaller surface-to-volume ratio than the flexural mode resonators of equal frequency, which is expected to lead to improved stability. (iii) The results were obtained for samples enclosed for rather imperfect vacuum (the residual pressure was $p \sim 5$ mbar). We anticipate that a significantly improved performance can be obtained using a more sophisticated encapsulation technology.

In general, the obtained results appear promising for achieving a reference-quality long-term stability in micoresonators. However, extensive further work is required for a full characterization. This includes a larger set of samples, mass-production suitable (water-level) encapsulation, careful consideration of possible packaging induced stress effects, and more extensive temperature and other operation environment cycling. We also note that the studied silicon microresonators exhibit the typical ∼28 ppm/K temperature dependence which has to be taken into account in potential frequency reference applications.

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References

[2] IEEE Std. 802.15.1; IEEE Std. 802.15.4.

Biographies

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Heikki Seppä received the M.Sc., Lic. Tech., and Dr. Tech. degrees from Helsinki University of Technology in 1977, 1979 and 1980, respectively. From 1976 to 1979, he was an assistant at the Helsinki University of Technology, working in the area of electrical metrology. He joined the Technical Research Centre of Finland (VTT) in 1979 and since 1989 he has been employed there as a Research Professor. In 1994, he was appointed Head of the Measurement Technology field at VTT Automation. Since 2002 he has acted as Research Director, VTT Information Technology. He has done research work on electrical metrology, in general, and on superconducting devices for measurement applications, in particular. He is doing research on dc-SQUIDs, quantized Hall effect, SET-devices, RF-instruments and microelectromechanical devices.

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