PERFORMANCE ENHANCEMENT AND RESTORATION OF MICROMECHANICAL RESONATORS VIA UV-OZONE TREATMENT

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ABSTRACT

An ultraviolet (UV)-ozone treatment process capable of atomic-scale surface cleaning has enabled the highest measured room temperature Q and frequency-Q product (f-Q) for on-chip *polysilicon* acoustic resonators to date while also demonstrating an ability to restore catastrophically contaminated devices to operational performance specs. Specifically, UV-ozone cleaning improved the Q of a heavily contaminated 61-MHz polysilicon wineglass disk resonator by over 282×, from a very-low 921 to a record-setting-high of 259,910. Post-release UV-ozone treatment further raises the Q of a 303.2-MHz polysilicon wineglass disk resonator by 30.5% to 89,172, yielding the highest measured f-Q to date of 2.70×10^{13} for *polysilicon* micromechanical resonators. Initial experiments seem to confirm that the high surface-to-volume ratio of micro and nano-scale structures amplifies susceptibility to surface-derived loss mechanisms. UV-ozone treatment also raised the Q of a 311.2-MHz AlN resonator from 789 to 2,574, providing evidence of a material agnostic benefit.

KEYWORDS

MEMS, quality factor, resonator, array, polysilicon, AlN, capacitive-piezoelectric, piezoelectric, VHF, UHF, ultraviolet, ozone, contamination, restore, yield.

INTRODUCTION

Having recently achieved Stratum 3E performance [1], the thought that vibrating MEMS-based timing oscillators might someday match the performance of low-end atomic clocks might no longer be as outrageous as it once was. Indeed, the combination of ultra-clean, reactive-sealed vacuum packaging [2] and extremely high resonator quality factor Q have reduced frequency drift to levels low enough to position MEMS-based oscillators as a preferred choice for 5G and next generation wireless applications, with obvious implications to future market share.

Very high Q in excess of 50,000 has been instrumental to performance gains, since high Q not only reduces close-to-carrier phase noise (hence improving short-term stability) but also reduces long-term drift by suppressing the temperature-dependence of the transistor circuits that sustain oscillation. High Q, however, is not enough, since stress relaxation, contamination, and structural changes also contribute to long-term drift. It is these phenomena that prevent vibrating MEMS oscillators from closing the long-term drift rate deficit relative to atomic clocks.

Pursuant to better understanding contamination-derived performance drift, this work employs atomic-scale cleaning via UV-ozone to study the degree to which minute contamination can affect the frequency and energy loss,

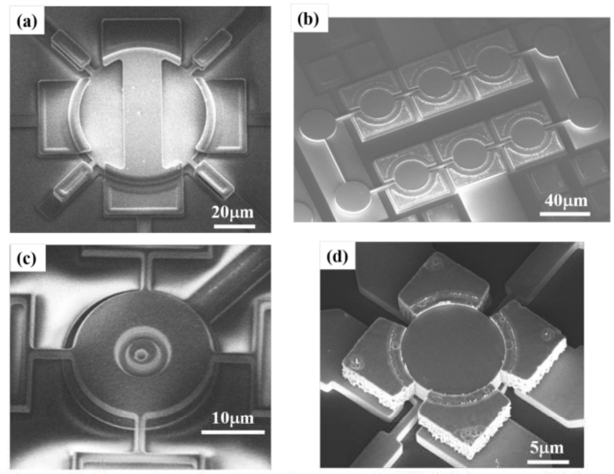

Figure 1: Scanning electron microscopic (SEM) image of (a) a 61-MHz polysilicon wineglass disk resonator, (b) a 199-MHz polysilicon disk array-composite resonator, (c) a 311-MHz AlN capacitive-piezoelectric disk resonator, and (d) a 303-MHz polysilicon wineglass disk resonator.

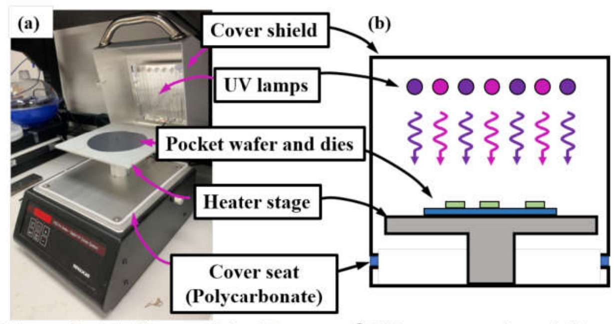

Figure 2: (a) Photo of the Novascan® UV-ozone tool and (b) an illustrative schematic of this tool in action.

i.e., Q, of various high frequency micromechanical resonators constructed in polysilicon and AlN, shown in Figure 1 [3] [4] [5]. This approach reveals that contaminants adsorbed over the span of a decade in a mostly indoor air environment can shift the frequency of a polysilicon wineglass disk resonator from 61.2515MHz to 61.2362MHz—a -249.8 ppm change—and its Q from 77,811 all the way down to 13,148—a more than 83.1% reduction. Shorter more controlled experiments further show that contaminant-induced Q reduction happens very quickly, within the span of hours, confirming long-held predictions that the high surface-to-volume ratio of micro and nano-scale structures amplifies susceptibility to surface-derived loss. In addition to a diagnostic tool, the results reported herein further reveal that UV-ozone treatment is uniquely effective in cleaning micromechanical resonators towards unprecedented performance, where UV-ozone makes possible a 303.2-MHz polysilicon wine-glass disk resonator with a Q of 89,172, yielding an f-Q product of 2.70×10^{13}, which

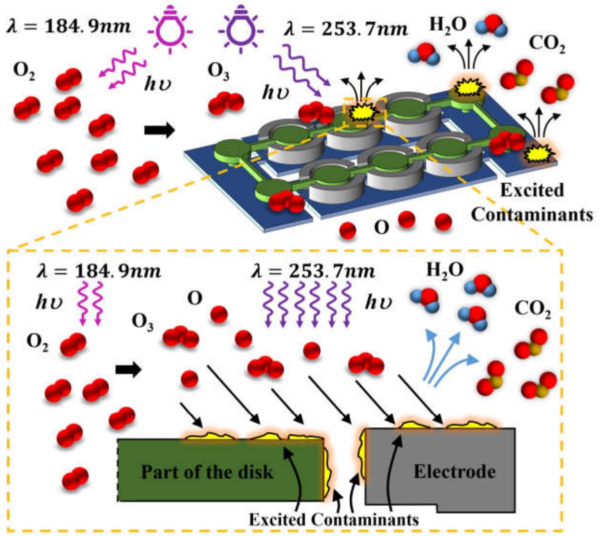

Figure 3: Illustrations summarizing the physical and chemical processes that drive UV-ozone cleaning of a typical micromechanical device. Here, the 184.9-nm line generates atomic oxygen and ozone from oxygen, and the 253.7-nm line excites organic molecules. Atomic oxygen and ozone react with energized organic molecules to form volatile molecules such as H_2O and CO_2 that escape in the gas stream.

is a record at this frequency for polysilicon. UV-ozone also raised the Q of a 311.2-MHz AlN resonator from 789 to 2,574, providing evidence of a material agnostic benefit.

UV-OZONE CLEANING

The use of UV-ozone to clean sample surfaces goes back to the early 1970's [6], when quartz resonator fabrication processes incorporated the method to clear out organics from surfaces and thereby maximize the aging stability of the resultant resonators. Curiously, while UV-ozone has been applied to MEMS structures [7], MEMS resonators have not benefitted from UV-ozone treatment to the same degree as quartz—something this work might remedy.

As shown in Figure 2, a typical UV-ozone cleaning system comprises a chamber containing oxygen and housing a low-pressure mercury lamp above a temperature-controllable substrate or device holder. When activated, the mercury lamp produces two UV lines: A 184.9-nm line that decomposes O_2 molecules to synthesize O_3 plus a highly reactive oxygen free radical; and a 253.7-nm line that decomposes O_3 to produce even more oxygen radicals. UV radiation also excites and dissociates contaminant molecules, principally organic ones, making them more susceptible to reaction with free O radicals. When organic contaminants are the target, CO_2 and H_2O are the reaction byproducts, both volatile at the treatment temperature (set by the sample holder) so both removable in the gas stream. Sufficiently long exposure to this reactive environment yields atomically clean surfaces. Figure 3 presents an illustrative summary of the pertinent physical and chemical processes over a disk array-composite resonator—one of the devices under test and pictured in Figure 1(b).

In addition to removing organics, the highly reactive O radical environment can also oxidize device surfaces, which might impose a limit on the length of exposure for a given sample.

DEVICE RESTORATION VIA UV-OZONE

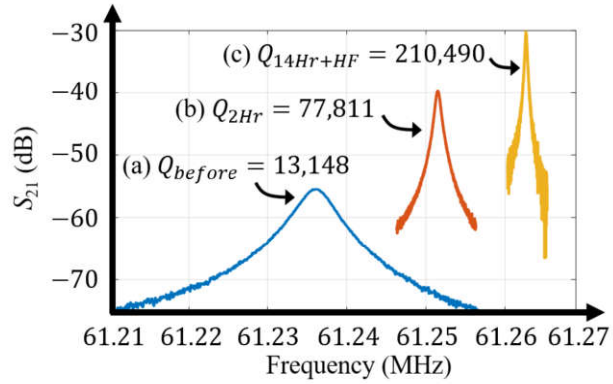

Figure 4: Measured S_{21} of a representative wine-glass disk (a) after 10-years in open air with a Q of 13,148, (b) after 2-hour UV-ozone with a Q of 77,811, and (c) after 14-hour UV-ozone + 1-min HF dip with a Q of 210,490.

A PSD Pro series digital UV-ozone system from Novascan®, pictured in Figure 2, provided the UV-ozone environments for this work. The UV lamps and wafer holder/heater are clearly visible in the photo. Since the tool takes a full wafer, the dies under test reside in a pocket wafer, i.e., with a pocket etched in its central region to hold smaller wafer pieces, during treatment.

Although the UV-ozone tool does provide a port to supply concentrated oxygen, this work simply uses oxygen in the air, in part to explore the efficacy of this approach for *in situ* possibilities. Thus, the times required for the results to follow are somewhat long but would likely be much shorter when using pure oxygen.

Short-Time-Span UV-Ozone Cleaning

The first die tested had been released almost 10 years ago and stored in room air. It houses forty 61-MHz wine-glass disks [3](each like that of Figure 1(a)) with 80.5-nm capacitive transducer gaps. Figure 4 presents the measured S_{21} parameter of one representative device before any cleaning, showing a poor Q of 13,148 indicative of substantial contamination over the years. A 2-hour UV-ozone cleaning (using room oxygen) improves the Q of this device by over 491% to 77,811. At the same time, the frequency of this device went from 62.2362 MHz to 61.2515 MHz, suggesting that this device aged -249.8 ppm in frequency over ten years due to contamination. This puts an exclamation point on the need for hermetic/vacuum packaging of MEMS resonators used for timing applications. Figure 5(a) and (b) provide a fuller picture of the degree of improvement by presenting distributions of measured Q for all forty devices before and after the 2-hour UV-ozone clean, respectively. Of note is the larger number of devices in (b) in the Q >100,000 bin. Although the granularity of the bins makes this hard to see, all device Q's improved with UV-ozone cleaning. Overall, after 2 hours of UV-ozone cleaning the average Q improved from 34,305 to 41,340, an increase of 20.5%.

Long-Time-Span UV-Ozone Cleaning

Attempts to further improve Q via much longer exposure to UV-ozone met with initial difficulty. Specifically, all forty of the 61-MHz wineglass disks stopped working after another 12-hour UV-ozone cleaning. Fortunately, a quick 1-minute HF dip successfully revived all 40

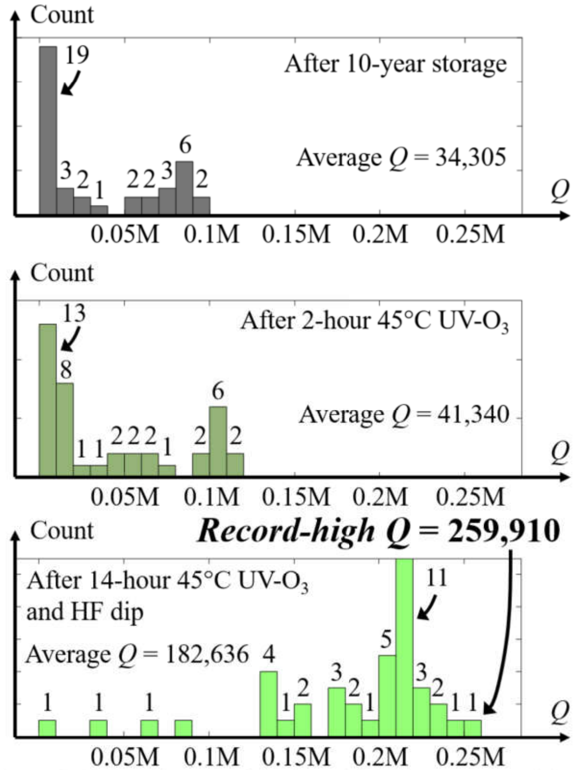

Figure 5: Q Distribution of the forty 61-MHz resonators (a) after 10 years in room air, (b) after 2-hour UV-ozone treatment and (c) after 14-hour UV-ozone treatment+ 1-min HF dip.

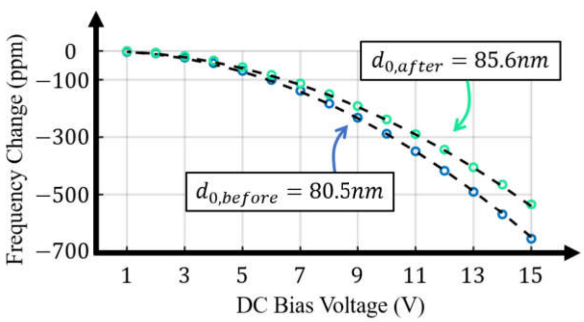

Figure 6: Measured frequency change vs. DC bias voltage to enable curve-fitted extraction of gap spacings before UV-ozone treatment (blue) and after (green).

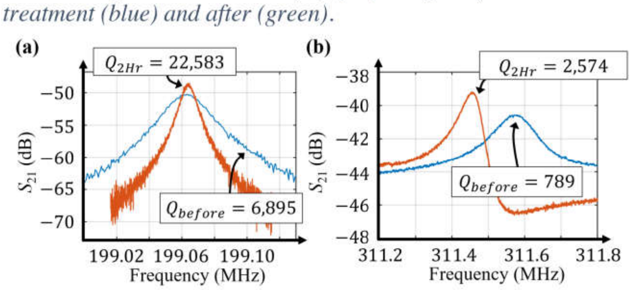

Figure 7:Measured S_{21} of (a) 199-MHz polysilicon disk array-composite resonator and (b) 311-MHz AlN capacitive-piezoelectric resonator before and after 2-hour UV-ozone treatments.

resonators, indicating that oxide growth during prolonged UV-ozone exposure was the likely culprit. After the HF dip, devices exhibited even higher Q's with the distribution as shown in Figure 5(c), where the majority of devices have Q >200,000 and only four have less than 100,000.

The plots of resonance frequency vs. DC bias voltage in Figure 6 provide further evidence of oxide growth during prolonged UV-ozone exposure. In particular, curve-fitting to extract the capacitive transducer gap spacing [3] before and after UV-ozone treatment reveals 80.5 nm and 85.6 nm, respectively—a 5.1 nm increase in the gap likely caused by oxidation of silicon and its removal.

Interestingly, one of the forty resonators posted a very-low Q of only 921 before cleaning and an ultra-high Q of 259,910 after UV-ozone plus HF dip, corresponding to an astonishing Q improvement of over 282 times. The end Q for this device is the highest measured room temperature value for an on-chip polysilicon acoustic resonator to date.

UV-Ozone on a Smaller-Gap Device

The second tested die housed a 199-MHz disk array-composite resonator [4] (like that of Figure 1(b)) with 39.2-nm capacitive transducer gaps released on Feb. 1, 2020. Figure 7 (a) presents the measured S_{21} parameter after 8-months in room air, showing a measured Q of 6,895. After a 2-hour UV-ozone treatment the Q increased by 228% to 22,583, the highest measured for this mechanical circuit.

UV-Ozone on a Piezoelectric Device

UV-ozone seems to also cure piezoelectric resonators. Specifically, the Q of a 311-MHz AlN capacitive-piezoelectric disk resonator (like that of Figure 1(c)) released on Feb. 16, 2020, after 8 months in room air, drops to 789. A subsequent 2-hour UV-ozone cleaning restores the Q to 2,574, as shown in Figure 7(b). The device, however, stops working after another 2-hour UV- ozone cleaning, possibly due to excessive AlN oxidation.

UV-OZONE FOR MANUFACTURING

While the previous section focus on device restoration yields important insights on the degree to which contamination debilitates micro-scale device performance, the record-setting Q's observed during restoration encourage incorporation of UV-ozone into device manufacture. This especially considering how contaminated tools, human-wafer interaction, etc., may introduce organic contamination to devices during the release process and hence lower Q.

Yet another die housing a 41.2-nm-gap 303.2-MHz polysilicon wineglass disk (like that of Figure 1(d)) was used to gauge the utility of UV-ozone for manufacturing. Figure 8 presents the measured frequency response of the resonator immediately after release and after a subsequent 2-hour UV-ozone treatment, showing a 30.5% improvement in Q from 68,332 to 89,172. This frequency and Q posts a record f-Q product of 2.70×10^{13} for polysilicon and interestingly is higher than the apparent theoretical limit of single crystalline silicon predicted by [8] (suggesting that the models used in [8] and other similar papers might require revision).

CONTAMINATION RATE INSIGHTS

Perhaps as interesting as the sheer performance enhancement afforded by UV-ozone cleaning are the physical insights obtainable. Specifically, the ability to clean micro- and nano-scale devices at the atomic scale invites experiments to better understand contamination processes.

For example, Figure 9 quantifies the rate at which contamination degrades devices by plotting the average Q of the forty devices on the 61-MHz wine-glass disk die as a function of time right after the UV-ozone cleaning process

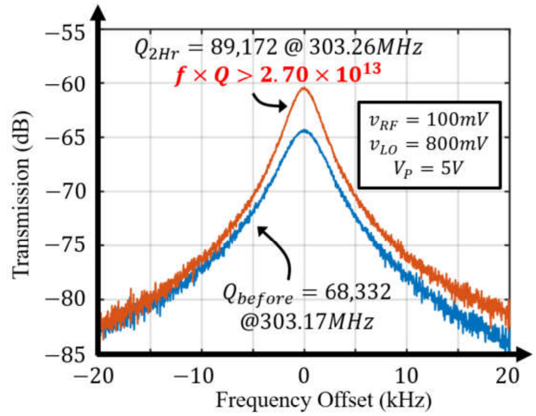

Figure 8: Measured S_{21} of the 303-MHz polysilicon wineglass disk resonator immediately after release (blue) and after 2-hour UV-ozone treatment (red).

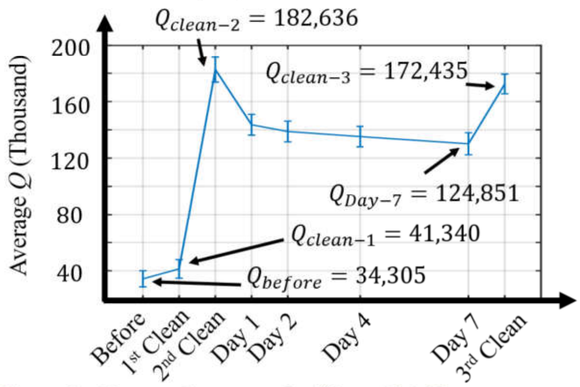

Figure 9: Measured average Q of forty 61-MHz resonators after 10-year storage in air, 1st clean (2-hour UV-ozone treatment), 2nd clean (12-hour UV-ozone treatment + 1min HF dip), 1-7 days of storage in under 0.5 mTorr vacuum, and after a 3rd clean (12-hour UV-ozone treatment + 1-min HF dip).

(and HF dip) and after 1-7 days of storage under 0.5 mTorr in a vacuum bulb that also housed several coaxial cables, which could be a source of outgassing. While perhaps not the most controlled experiment, measurements do reveal that the average Q drops quickly at first—by 20.6% in 1 day—but then more slowly by 28.7% over the next 6 days. This perhaps indicates a contamination mechanism where contaminants adsorb rapidly at first but then saturate, perhaps over a monolayer, after which contamination proceeds more slowly.

Clearly, UV-ozone cleaning now provides ample opportunity for future study of contamination processes in micro- and nano-scale mechanical resonators and their effect on performance.

CONCLUSION

The ability of UV-ozone treatment to not only restore contaminated MEMS resonators devices but do so with record-setting Q performance is a clear game-changer for applications that employ such devices. This especially goes for MEMS-based timing, but also gyroscopes, sensors, and any application that places a premium on frequency stability. For example, UV-ozone treatment might be the key to attaining ultra-clean packaged environments for MEMS devices without the need for *in situ* reactive sealing. Perhaps even more interesting than sheer performance are the insights to come when researchers begin to employ UV-ozone atomic-scale cleaning to explore the behavior and resilience (or lack thereof) of micro- and nano-scale devices, resonant and non-resonant, under various contaminant environments.

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