# Study on Surface Roughness Improvement of Fused Quartz After Thermal and Chemical Post-Processing

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***Abstract*—We present a study on the effects of thermal and chemical post-processing on the surface quality of Fused Quartz, a preferable material for vibratory gyroscopes. In this study, we included the effects of thermal reflow, potassium hydroxide (KOH) etching, Buffered Oxide Etching (BOE), 10:1 hydrogen fluoride and hydrogen chloride solution (HF/HCl) etching, and RCA-1 surface treatment. We concluded that thermal reflow of fused quartz at 1300°C for one hour achieves the best result, followed in effectiveness by the RCA-1 surface treatment, while KOH etching, BOE, and HF/HCl etching are shown to deteriorate the surface quality. The effects of thermal reflow were studied for 2-D and 3-D geometries. The improvement in surface roughness of microstructures was shown to directly correlate to improvements in the quality factor.**

***Keywords—MEMS; fused quartz; surface roughness; quality factor; vibratory gyroscope.***

## I. INTRODUCTION

Quality factor is a critical parameter to enhance performance of vibratory inertial MEMS devices [1]. Better in-run noise performance, such as bias stability and Angle Random Walk (ARW) can be achieved with higher quality factor of resonating structure. Obtaining the quality factor on the level required to achieve the navigation grade performance remains to be a challenge due to factors such as viscous damping, ThermoElastic Dissipation (TED), intrinsic material loss, surface loss, and anchor loss. Total quality factor of the vibratory structure can be calculated as in (1) and is dominated by the loss mechanism with the lowest quality factor component.

$$Q_{total}^{-1} = Q_{viscous}^{-1} + Q_{TED}^{-1} + Q_{surface}^{-1} + Q_{anchor}^{-1} + Q_{others}^{-1} \quad (1)$$

Viscous damping can be eliminated by packaging and vacuum sealing of devices. Anchor loss is related to the energy dissipation through anchor and can be mitigated by proper design of the structure to balance the total dynamic reaction force and torque. TED is largely related to the material properties, such as the Coefficient of Thermal Expansion (CTE), and also to the geometry of devices. Current MEMS materials, such as single-crystal silicon, have a relatively high CTE, on the level of 2-3ppm/°C. Fused quartz, however, due to its low CTE (0.5ppm/°C) and isotropic mechanical properties, is a preferable material for vibratory MEMS devices, such as mode-matched gyroscopes. Large-scale, centimeters in diameter, fused quartz gyroscopes reported the quality factor on the order of 25 million [2], inspiring the development of small-scale, millimeters in diameter, fused quartz inertial sensors.

As dimensions of MEMS sensors are scaled down, the surface-to-volume ratio increases, and thus the effect of surface loss on the quality factor becomes a dominant factor for small-scale sensors. As reported in [3], the quality factor of Hemispherical Resonator Gyroscope (HRG) is strongly dominated by surface loss and has an inverse relationship to the surface-to-volume ratio. The achievement of high surface quality is a priority for making the progress in performance improvement of inertial sensors. In previous studies, 10:1 HF/HCl etching is reported to create a smoother surface on Pyrex and soda-lime-glass than the HF etching [4]. BOE is reported to improve the surface of fused quartz devices [5]. However, no systematic studies and comparative analyses have been reported in the literature. This paper intends to fulfill this gap.

We present a study on the effects of thermal and chemical post-processing on the surface quality improvement of fused quartz. Our study includes the effects of HF etching, KOH etching, RCA-1 surface treatment, which are common in the fabrication of fused quartz wineglass gyroscopes [6], as well as thermal reflow, BOE, and 10:1 HF/HCl etching, showing that some treatments improve while others deteriorate the surface quality. The improvement in surface roughness of micro-structures was shown to directly correlate to improvements in quality factor of fused quartz wineglass resonators.

## II. EXPERIMENTAL PROCEDURE

In this study, the effects of thermal and chemical post-processes were initially tested on blank fused quartz wafers. Then, the effects of thermal reflow were tested on curved 3-D fused quartz structures to show that the conclusions derived from blank samples are applicable to curved surfaces. Finally, fused quartz wineglass resonators [7] with different surface quality were characterized. The quality factor was measured and compared to demonstrate the direct correlation between surface roughness and quality factor.

Atomic Force Microscope (AFM) from Pacific Nanotechnology (Nano-R) was used to measure the surface roughness of samples. The scan area was 10μm × 10μm for blank fused quartz samples and all samples were measured at three different points. Samples were cleaned by standard solvent cleaning before each scan. The AFM was run in a close contact mode, using a 10 nm radius probe tip (Agilent U3120A). For 3-D wineglass shell resonators, the samples were diced into smaller pieces to make sure no other parts of the sample except for the point being measured would be in contact with the AFM tip.

Quality factors of the wineglass resonators were measured from the frequency sweep. The resonators were actuated piezoelectrically by a piezo stack attached to the resonator's stem. The amplitude of vibration was measured by Laser Doppler Vibrometry (LDV). All resonators were cleaned by standard solvent cleaning and RCA-1 cleaning before all tests. The devices were operated in a vacuum chamber under pressure of 20μTorr, so that the viscous damping is completely eliminated [6].

## III. RESULTS AND DISCUSSION

### A. Chemical Post-Processing

A fused quartz wafer goes through many chemical processes before it is fabricated into a wineglass resonator [6, 7]. All these processing steps have an influence on the surface quality of samples. Typical processes include 48% HF etching, 45% KOH hard mask removal, and RCA-1 cleaning. Some processes also include 10:1 HF/HCl etching and BOE. The effects of all these treatments on the surface quality of fused quartz were investigated in this study. Each treatment was applied to samples for the time duration that is typical for the fabrication process of wineglass resonator. The results are summarized in Table I.

Table I shows that both KOH etching and BOE deteriorate the surface of fused quartz, and HF etching creates a lower surface roughness than 10:1 HF/HCl solution, while RCA-1 surface treatment improves the surfaces of fused quartz samples by reducing the averaged surface roughness from 6.3nm Sa to 4.7nm Sa. To uncover the active components of RCA-1 solution and understand mechanisms of the effect, three fused quartz samples with the same surface quality were placed into RCA-1 solution (volume ratio of 1:1:5 for 27% $NH_4OH$, 30% $H_2O_2$, and DI water), $H_2O_2$ solution (0:1:6), and $NH_4OH$ solution (1:0:6), respectively. The reaction temperature was controlled at 80°C and the reaction time was 20 minutes for all three samples. The results are shown in Table II.

TABLE I. EFFECTS OF TREATMENTS ON SURFACE ROUGHNESS

| **Sample status** | **Surface roughness Sa* (nm)** | | | |
|---|---|---|---|---|
| | ***Point 1*** | ***Point 2*** | ***Point 3*** | ***Averaged*** |
| Original | 25.7 | 25.2 | 22.6 | 24.5 |
| 10min KOH | 50.5 | 47.7 | 47.8 | 48.7 |
| Original | 1.2 | 1.1 | 1.2 | 1.2 |
| 30s BOE | 5.4 | 5.2 | 5.5 | 5.4 |
| 5h HF | 1.7 | 1.3 | 1.0 | 1.3 |
| 5h HF/HCl | 10.7 | 12.0 | 9.4 | 10.7 |

*Sa *denotes the arithmetic average of a height function of a surface*

TABLE II. EFFECTS OF RCA-1 AND ITS COMPONENTS

| **Sample status** | **Surface roughness Sa (nm)** | | | |
|---|---|---|---|---|
| | ***Point 1*** | ***Point 2*** | ***Point 3*** | ***Averaged*** |
| Original | 7.5 | 5.6 | 5.8 | 6.3 |
| RCA-1 | 4.3 | 5.1 | 4.8 | 4.7 |
| $H_2O_2$ | 7.5 | 6.1 | 6.2 | 6.6 |
| $NH_4OH$ | 6.2 | 5.5 | 5.7 | 5.8 |

The results show that the effect of RCA-1 on the surface quality of fused quartz sample is mainly due to $NH_4OH$, since the averaged surface roughness is reduced from 6.3nm Sa to 5.8nm Sa. $H_2O_2$ alone does not improve the surface quality, but enhances the smoothening effect of $NH_4OH$. According to [8], the $SiO_2$ etching rate increases with increasing $NH_4OH/H_2O_2$ ratio and with increasing concentration of $NH_4OH$. This result suggests that the surface roughness of sample may become worse if a higher etching rate is applied for certain etchants.

### B. Thermal Post-Processing

#### 1) Determination of reflow temperature and duration

Fused quartz is an amorphous material that does not have a precisely defined melting temperature. As the temperature increases, the material becomes soft and its viscosity decreases. If the temperature of treatment is high enough, the surface tension of fused quartz can overcome viscosity and therefore minimize the surface area, like in a liquid. Using the reflow phenomenon, the surface roughness can be improved. On the one hand, the reflow temperature has to be high enough, so that the surface tension can overcome viscosity and smoothen the surface. On the other hand, the temperature cannot be too high, so that the sample does not deform and lose its axial symmetry due to gravity.

Fused quartz wineglass resonators were reflowed for 30 minutes at different temperatures, ranging from 1100°C to 1400°C, with 1300°C turned out to be the highest temperature at which no obvious deformation was observed. Therefore, the reflow temperature was set to be 1300°C for experiments.

The change of surface roughness over time was studied. Thermal reflow at 1300°C was applied on blank fused quartz samples with the same surface roughness for different time durations. The results are shown in Fig. 1. The surface roughness is reduced in general with increasing the time of treatment and is only improved moderately after 30 minutes. It can be concluded from Fig. 1 that the reflow time of 1 hour is sufficient, reducing the averaged surface roughness from 24.5nm Sa to 1.9nm Sa.

#### 2) Analysis of results on blank samples

The averaged surface roughness did not change significantly during the first five minutes, but was greatly reduced with an extended duration of the experiment. To understand the process during the first five minutes, the Power Spectral Density (PSD) was analyzed. PSD of a surface is related to the 2-dimensional Fourier transformation of the surface height function and it contains more information than an averaged surface roughness. PSD is expressed as

$$P(\mathbf{k}) = \frac{1}{(2\pi)^2}\left|\int h(\mathbf{x}) e^{-i\mathbf{k}\cdot\mathbf{x}} d\mathbf{x}\right|^2, \qquad (2)$$

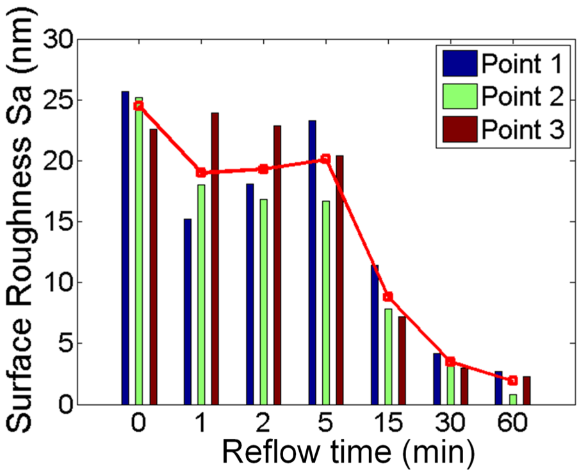


Fig. 1. Surface roughness changes over reflow time. Each sample was measured at three different points and the red line represents an average value. Averaged surface roughness is reduced in general with increasing time, but the smoothening effect is not obvious during the first five minutes.

where k is the wave number, x is the position vector, and h(x) is the height function of the surface. Fig. 2 shows the PSD of original surface and surfaces after reflowing for 1min, 2min, and 5min. It shows that these surfaces can be modeled as self-affined [9] and surface roughness exponents α can be extracted to characterize the surfaces. Roughness exponent characterizes the short-range roughness of a self-affined surface. It ranges from 0 to 1. A small value of α implies a rougher local surface, Fig. 3 [9]. For self-affined surfaces, the PSD and surface roughness exponent have a relation

$$P(k) \propto k^{-2-2\alpha}, \quad for\ k \gg \xi^{-1}, \tag{3}$$

where ξ is the lateral correlation length of the surface. Fig. 2 shows that the surface roughness exponent increases from 0.458 to 0.671, indicating that the short-range roughness is improved by reflow, although the general averaged surface roughness does not change.

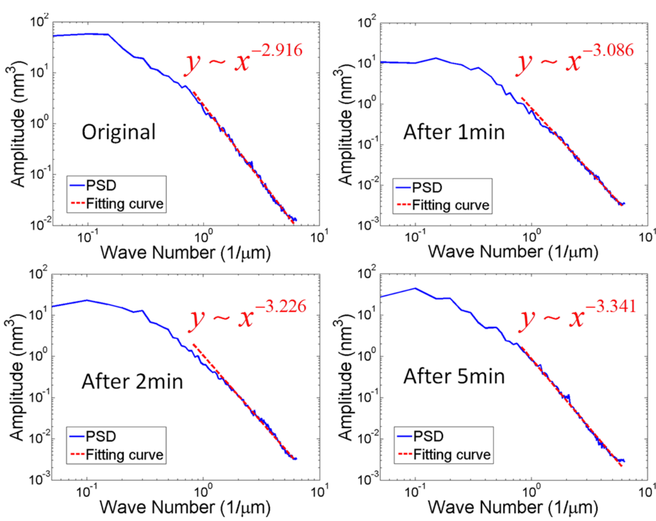


Fig. 2. Power spectral densities of the original surface and surfaces after reflowing for 1min, 2min, and 5min. The results show that these surfaces can be modeled as self-affined. Surface roughness exponents increase from 0.458 to 0.671, indicating that surface quality is improved by the thermal reflow.

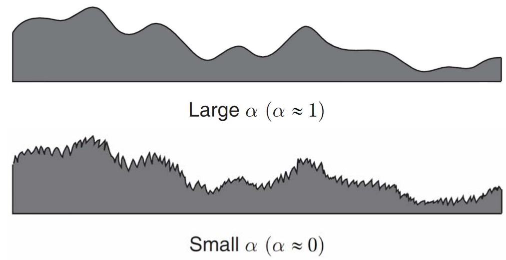


Fig. 3. A comparison of the local surface morphology for similar surfaces with different values of α. A smaller value of α implies a rougher local surface, where α lies between 0 and 1.

Histograms of the original surface and surfaces after reflowing for 5min, 15min, and 60min are shown in Fig. 4, which is a characterization of the long-term surface morphology. The shape and position of the distribution peak in histogram do not change in the first five minutes and only very high elevation regions of the surface are affected. This confirms that only a short-range roughness improvement happens during this time period. After the first five minutes, the peak becomes more concentrated and shifts to the left, indicating a better surface quality.

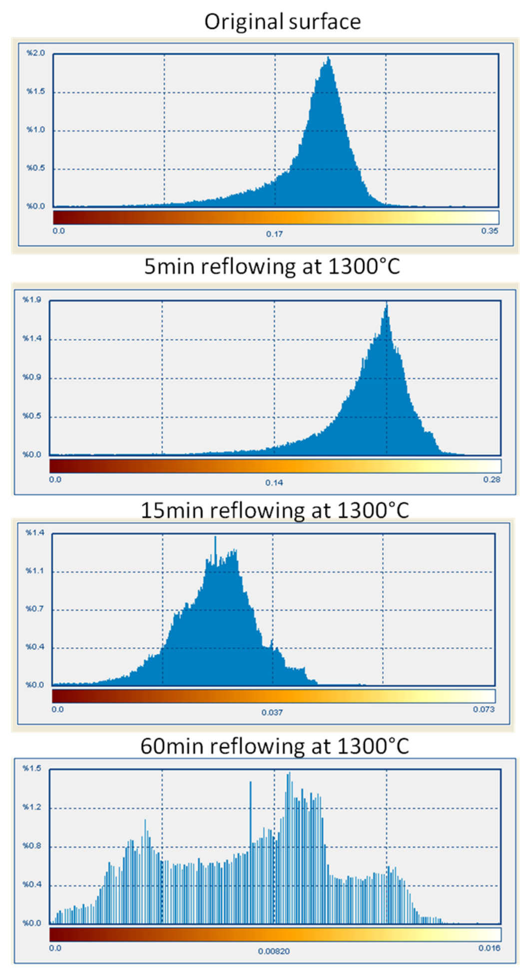


Fig. 4. Histograms of the original surface and surfaces after reflowing for 5min, 15min, and 60min, showing an improvement of long-term surface roughness. The X-axis is height and the Y-axis is percentage of the area at a corresponding height.

*3) Thermal reflow applied to 3-D structures*

All the previous results are reported for blank fused quartz samples. To show that the same effects can be observed on 3-D wineglass structures, the surfaces were measured before and after the thermal reflow. Two glassblown samples from the same batch were tested before and after the 1 hour reflow at 1300°C, respectively. The results are shown in Fig. 5. Averaged surface roughness was reduced from 1.9nm Sa to 0.19nm Sa. The same smoothening effect was observed in the 3-D structures, implying that all the conclusions above are applicable to curved surfaces. Besides, surface height distribution is lower and more concentrated, indicating a better surface quality after the thermal reflow.

### C. Surface Roughness and Quality Factor

One of the goals of this study is to further improve the quality factor of fused quartz vibratory gyroscopes, such as wineglass rate integrating gyroscopes. In this section, the quality factors of resonators with different surface roughness were measured, showing a direct correlation between the improvement in the quality factor and better surface quality.

Three fused quartz wineglass resonators were tested in this study. First, the resonators were characterized and the quality factors were measured. Then, 10 minutes of KOH etching was applied to the resonators to roughen the surface and the quality factors were measured again. Then, the resonators were thermally reflowed at 1300°C for 1 hour to improve the surface roughness and the quality factors were measured for the third time. Results are shown in Table III. A direct correlation between the quality factor of resonators and surface roughness can be concluded. The decrease of quality factor related to KOH etching was 17,000 on average and the increase of quality factor due to thermal reflow was about 6,000. It indicates that a large part of the roughening effects due to KOH etching can be compensated by thermal reflow. All other loss mechanisms were remained the same, leading to the conclusion that changes in quality factor were due to surface losses.

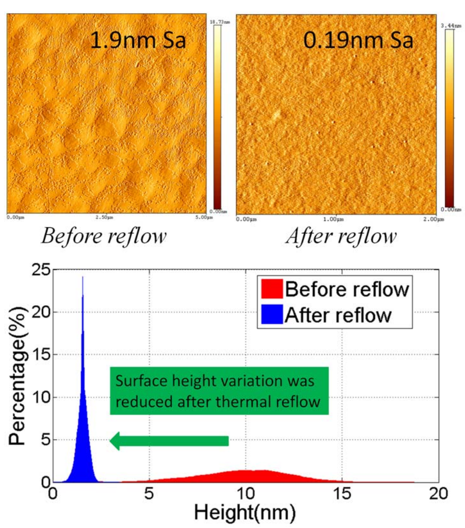


Fig. 5. AFM images of the surface of 3-D fused quartz structures and histograms before and after reflow. The averaged surface roughness is reduced from 1.9nm to 0.19nm and surface height distribution is lower and more concentrated, indicating a better surface quality.

TABLE III. QUALITY FACTORS AFTER TREATMENTS

| Sample number | Quality factor | | |
|---|---|---|---|
| | ***Original*** | ***After KOH*** | ***After reflow*** |
| 1 | 49.9k | 25.6k | 30.2k |
| 2 | 60.1k | 42.5k | 46.3k |
| 3 | 16.1k | 6.3k | 15.8k |

## IV. CONCLUSIONS

The effects of chemical and thermal post-processing on the surface quality of fused quartz samples were studied. Thermal reflow at 1300°C for 1 hour showed the best result, reducing the averaged surface roughness from 24.5nm Sa to 1.9nm Sa. RCA-1 surface treatment improved the surface quality, but insignificantly.

A direct correlation between a better surface quality and a higher quality factor of the device has been established, indicating that the thermal reflow and RCA-1 surface treatment can increase the quality factor of fused quartz devices.

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## REFERENCES

[1] M. Weinberg, R. Candler, S. Chandorkar, J. Varsanik, T. Kenny, and A. Duwel. "Energy Loss in MEMS Resonators and The impact on Inertial and RF Devices". *Solid-State Sensors, Actuators and Microsystems Conference (TRANSDUCERS),* 2009, pp. 688-695

[2] D. M. Rozelle, "The Hemispherical Resonator Gyro: From Wineglass to the Planets," *Proc. 19th AAS/AIAA Space Flight Mechanics Meeting,* 2009, pp. 1157-1178.

[3] E. J. Loper, D. D. Lynch, and K. M. Stevenson, "Projected Performance of Small Hemispherical Resonator Gyros," in *PLANS '86 – Position Location and Navigation Symposium*, 1986, pp. 61-64

[4] C. Iliescu, J. Jing, F. E. Tay, J. Miao, and T. Sun. "Characterization of masking layers for deep wet etching of glass in an improved HF/HCl solution." *Surface and Coatings Technology* 198.1 (2005), pp. 314-318.

[5] J. Cho, T. Nagourney, A. Darvishian, B. Shiari, J. Woo, and K. Najafi. "Fused silica micro birdbath shell resonators with 1.2 million Q and 43 second decay time constant". *Hilton Head Workshop,* 2014, pp. 103-104.

[6] D. Senkal, M. J. Ahamed, M. H. A. Ardakani, S. Askari and A. M. Shkel, "Demonstration of 1 Million Q-Factor on Microglassblown Wineglass Resonators With Out-of-Plane Electrostatic Transduction," *IEEE/ASME Journal of Microelectromechanical Systems*, 24 (1), February 2015, pp. 29-37.

[7] D. Senkal, M. J. Ahamed, A. A. Trusov, A. M. Shkel, "High Temperature Micro-glassblowing Process Demonstrated on Fused Quartz and ULE TSG" *Sensors and Actuators A: Physical*, 201, 2013, pp. 525-531.

[8] H. Kaigawa, K. Yamamoto, and Y. Shigematsu. "Etching of thermally grown SiO2 by NH4OH in mixture of NH4OH and H2O2 cleaning solution." *Japanese journal of applied physics* 33.7R (1994): 4080.

[9] M. Pelliccione, and T. M. Lu. "Evolution of thin film morphology: modeling and simulations". Vol. 108. Springer Science & Business Media, 2008.