Glass-Blown Pyrex Resonator with Compensating Ti Coating for Reduction of TCF

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Abstract—**This paper demonstrates that the Thermal Coefficient of resonant Frequency (TCF) of a micro glass-blown Pyrex spherical resonator can be substantially reduced by the application of a titanium (Ti) coating. Finite Elements Analysis (FEA) is used to demonstrate that the temperature dependence of the Young's modulus of the shell material is the dominant parameter affecting the TCF of the resonator, clearly suggesting the use of a metallic compensating layer. Experimental characterization demonstrates that the TCF of a Pyrex glass-blown resonator is reduced by 70% (from 73ppm/°C to 24ppm/°C) by the application of a 1.33um thick layer of Ti. It is predicted by FEM that for a Ti layer thickness on the order of 2.5μm, the TCF will fall below 10ppm, an acceptable value for high performance resonators. This investigation is a step forward in the quest to employ the desirable properties of micro-blown resonators, such as high symmetry, manufacturing tolerances and environmental robustness.**

Keywords—*glassblowing, resonators, TCF compensation, dynamic MEMS.*

I. INTRODUCTION

Micro-Electro-Mechanical Systems (MEMS) resonators are desirable for a wide variety of applications, including signal processing, timing, frequency control, and inertial sensing [1]. Several silicon MEMS resonators are currently on the market [2-3]. The vast majority of MEMS resonators are fabricated utilizing silicon as a structural material and using photolithography and DRIE techniques for defining the features. The dimensional resolution of these techniques is limited by fabrication imperfections introduced by etching, such as DRIE-induced scalloping, surface roughness, and the intrinsic limits of the aspect ratio of features. As a result, the fabrication of highly symmetric, frequency-matched devices with exceptionally high quality factor becomes extremely challenging. These factors motivate the investigation of alternative fabrication approaches that allow the development of non-planar MEMS resonator architectures with increased symmetry, reduced roughness of structures, and increased aspect ratios—all accomplished simultaneously.

Recently, there has been significant interest in the development of 3-D MEMS spherical and hemispherical resonators for use in timing and inertial sensing applications. Photolithography and DRIE based approaches have been used to fabricate silicon oxide hemispherical resonators using silicon molds, resulting in quality factors (Q) as high as 20,000 at a resonant frequency of 22kHz [4]. The same fabrication approach has been demonstrated to build polysilicon resonators for inertial application, with Q's around 8000 at a resonant frequency of 416kHz [5]. Plastic deformation of metallic glasses to achieve spherical structures has been explored by using a blow-molding technique [6]. The use of low Thermo-Elastic Damping (TED) materials, such as Fused Silica (FS), as structural material has also being explored; Q factors above 100,000 at frequencies around 10kHz have been demonstrated [7]; processing of these materials requires temperatures in excess of 1,600°C.

A highly parallel glass-blowing process developed in the UC Irvine Microsystems Laboratory allows shaping of fused silica glass to create highly symmetric structures with resonant frequencies in the range from 1 kHz to 1MHz [8]. Although high Q factor (several thousands) and excellent symmetry was demonstrated (resulting in frequency splits as low as 0.15Hz), dimensional constraints of the hemispherical shapes may not lead to the highest fxQ product and environmental robustness. By fabrication of quasi-spherical structures using micro glass-blowing techniques [9], we are able to build spherical resonators with integrated electrodes and with low order resonant frequencies about 1MHz for resonators [10] and inertial sensors [11].

The high temperature dependence of the resonant frequency (TCF) in low TED glass resonators (fused quartz or borosilicate glass) is a major performance-limiting factor, making it impossible to achieve acceptable frequency stability [12], as practical frequency stability <100ppm over the range of operating conditions is required by most practical applications [13]. Several approaches exist to compensate for the frequency drift. They can be divided in two categories: (i) resonator-level solutions (engineering of Young's modulus [14], doping [15], compensation utilizing electrostatic spring softening [16], and temperature stabilization by ovenization [17]), and (ii) oscillator concepts based on frequency synthesis [18] and impedance loading [19]. Most often, several approaches are combined together to maximize temperature stability.

In this paper, we investigate the application of titanium coating on spherical glass-blown resonators for temperature compensation. Figure 1 shows a spherical resonator surrounded by a toroidal electrode structure. In the proposed design, the 3D electrodes are used to exert the electrostatic force to drive the mechanical resonance of the central sphere and to read-out its velocity. The application of the Ti layer is anticipated to
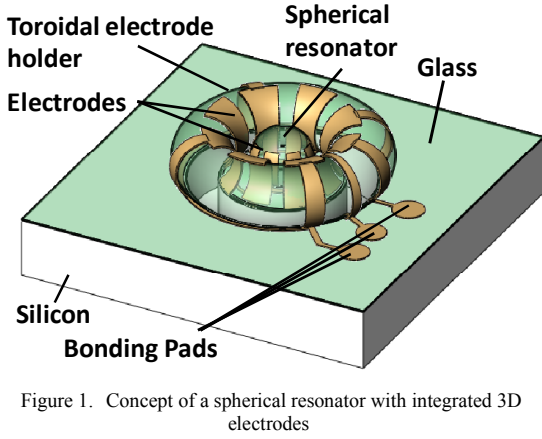
Figure 1. Concept of a spherical resonator with integrated 3D electrodes
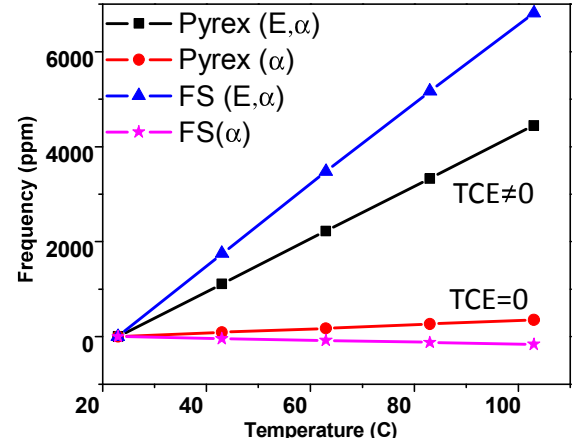

Figure 2. Modeled TCF for Pyrex and fused silica. Results shows the effect of the temeperature coefficient of Young's modulus

simultaneously control the TCF of the glass and provide the electrical conductive layer required to electrically bias the central spherical resonator

II. TCF MODELING

The Temperature Coefficient of resonant Frequency (TCF) determines the change of the resonant frequency of MEMS devices due to environmental variations in temperature. Three factors contribute to the frequency change of a glass-blown resonator: (i) dimensional changes in the resonator due to thermal expansion; (ii) thermal stresses resulting from Thermal Expansion Coefficient (CTE) mismatch between the glass and the silicon substrate, and (iii) intrinsic variations in Young's modulus of the glass with temperature (Temperature Coefficient of Young's Modulus, or TCE).

The thermo-mechanical behavior of the spherical resonator has been modeled with Finite Elements Analysis (FEA) using a commercial package COMSOL Multiphysics. The resonator is modeled as a 480μm radius sphere with 92% sphericity and non-uniform shell thickness (15um at the top, tapering to 40 μm near the anchor point). The silicon substrate is not included in the modeling. The thermo-mechanical properties of the materials used in modeling have been extracted from the COMSOL library and are reported in Table I. To investigate the dependence of both the TCE and CTE parameters on the TCF, the n=2 mode of a Fused Silica and Pyrex spherical resonator has been modeled and simulated.

Figure 2 shows the resonant frequency change of the n=2 mode of a spherical resonator due to the variation in temperature. For Pyrex and Fused Silica, two modeling experiments have been performed. In the first experiment we

TABLE I. THERMAL POPERTIES OF MATERIAL (OBTAINED FROM [20] AND THE MATERIAL LIBRARY BUILT IN COMSOL MULTIPHYSICS)

Material	Young's Modulus (@24°C)	CTE, α	TCE (ppm/C)
Fused Silica	73GPa	0.25ppm/C	+175
Borosilicate	60GPa	3.05ppm/C	+105
Silicon	170GPa	2.6ppm/C	-63
Pyrex 7740	63GPa	3.05ppm/C	+105
Titatium	128GPa	8.6ppm/C	-328

ignored the temperature dependence of the Young's modulus (TCE=0 in Figure 2), highlighting the TCF contribution from CTE dimensional changes. In the second modeling experiment we included the effect of both the TCE and the CTE. Two key results emerged: (a) the contribution of the temperature coefficient of the Young's modulus is the dominant effect, resulting in a large positive TCF for both materials; (b) when all factors are included, the TCF of fused silica (85.1ppm/°C) is substantially larger than that of Pyrex (55.5ppm/°C).

III. ENGINEERED TCF

We explored the composite Young's modulus to compensate the TCF. This technique is based on the fabrication of composite structural hybrid structures, encompassing at least two layers of different materials with opposite TCE. Metals are perfect candidates for compensating layers, as in addition to their negative TCE, metals have large electrical conductivity and hence can be used as electrode structures. Among all metals, titanium presents one of the highest negative TCE
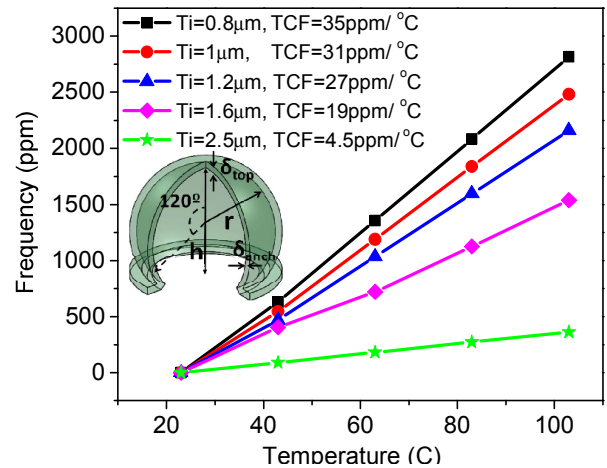

Figure 3. Modeling results of the effect of the titanium compensation layer on r=480μm, h=850μm, δ_{top}=15μm, δ_{anch}=40μm Pyrex spherical shells, where r, h, δ_{top} and δ_{anch} are radius, height, thickness of the top of the shell and thickness of the shell near to the anchor point, respectively.
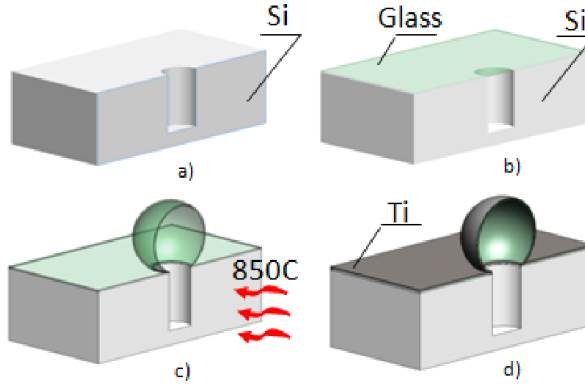
Figure 4. Fabrication flow of the temperature compensated spherical resonator, excluding a step for electrodes patterning.

Figure 6. In air non-contact characterization set up.

(Table I), allowing significant compensation with minimum thickness. Figure 3 shows the simulation results for titanium-coated Pyrex resonators. All geometric parameters are the same for all modeling results. Notice that 0.8um of Ti reduces the TCF of the resonator from 55.5ppm/°C to 35ppm/°C, and a TCF of less than 10ppm/°C can be achieved by using 2.5um of Ti.

A detailed description of the fabrication process for micro-glass-blown resonators can be found in [10]. Figure 4 shows a simplified scheme of the fabrication process that includes the compensation metal layer deposition. A silicon wafer is DRIE etched, (a), and subsequently anodically bonded to a 200um-thick Pyrex wafer, (b). Then, a glass-blowing step is performed, (c), and finally a Ti layer is sputtered (using a Denton Sputterer), (d). Figure 5 shows the resonator before and after the sputtering step, (a) and (b) respectively; and a SEM image of the cross-section after the sputtering step, (c).

IV. CHARACTERIZATION

The characterization set-up is shown in Figure 6. The device is mounted on a rotary stage and placed on a hotplate. The vibration is induced using a piezo-disk. Temperature of the die is monitored using a contact thermocouple, while the temperature of the glass bubble is monitored by an infrared camera. A Polytec MSA-500 vibrometer was used to measure the velocity of vibration of the spherical resonator in response to piezoelectric excitation in a direction perpendicular to the substrate. Four different devices have been characterized, with different thicknesses of the titanium layer. Although all devices were otherwise nominally identical, small deviations in radius resulted in slight changes in their resonant frequencies (Table II).

Figure 7 shows the experimental results of the frequency drift for the n=2 mode for 4 different spherical resonators. The
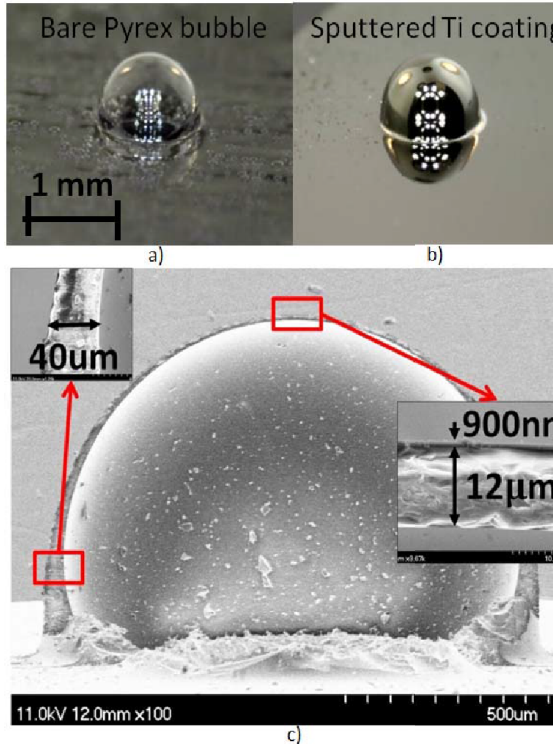

Figure 5. Optical images of the spherical resoantor a) before and b) after the Ti coating deposition. c) shows a cross section SEM image of a coatied spherical resoantor..
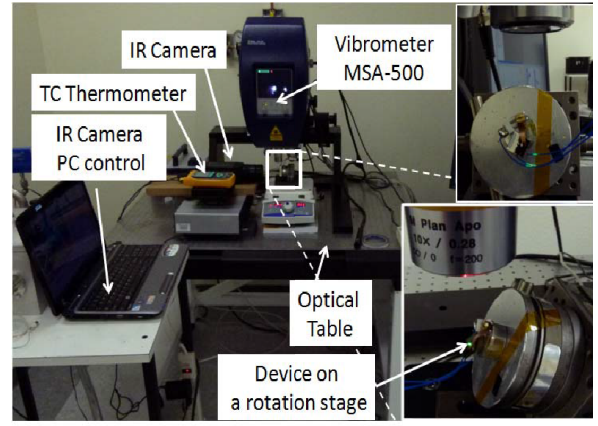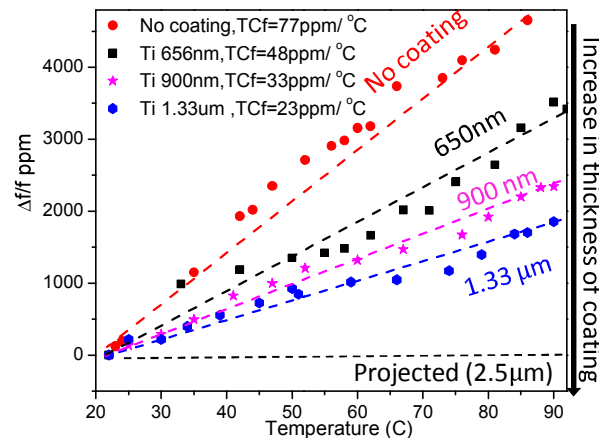

Figure 7. Experiemental results of frequency variation inspherical resonator with temeperature. The TCF values have been extracted form the measurements.

Device	Ti coating(um)	Frequency (Hz) (T=23CK)		TCf(α,E) (ppm/C)	Q_{air}	
		N=2	N=3		N=2	N=3
1	0	1.577700E6	1.785236E6	73±2.6	270	580
2	0.656	1.49E6	1.662774E6	48.5±1.6	285	518
3	0.900	1.615274E6	1.820801E6	33±1.08	179	468
4	1.33	1.455039E6	1.617207E6	23±1.3	298	514

temperature range varies from room temperature to ~90°C. The experimental TCF for a non-coated resonator was 73ppm/°C, 32% higher than FEA prediction (55ppm/°C). This discrepancy is attributed to the TCE mismatch between the silicon substrate and the Pyrex shell. The characterization results showed a clear reduction of the TCF for the spherical resonator by increasing the thickness of Ti metal. In particular, a reduction of 70% was achieved by using 1.33um film of metal.

Table II shows a summary of the characterization results. Importantly, in all devices that have been characterized, the Q factor does not decrease significantly upon application of the titanium coating. The study of the effect on Q is outside the scope of this paper.

V. CONCLUSIONS

This work demonstrates a viable approach for permanent TCF reduction in micro glass-blown Pyrex spherical resonators for high frequency applications. A metal coating fabrication step has been added at the end of an established micro glass-blowing fabrication process. Experimental results demonstrate the use of Ti as compensation layer. TCF reduction from 73ppm/°C to 25ppm/°C, without affecting the Q-factor of the n=2 resonant mode was demonstrated with an application of 1.33um Ti coating. The use of Ti mitigates the risk of dramatic temperature induced frequency drift on glass-based mechanical structures, and provides a convenient path for defining electrodes for electrostatic actuation.

ACKNOWLEDGMENTS

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