ABSTRACT
We report, for the first time, on the successful fabrication and operational characterization of electroplated Invar Micro-Hemispherical Shell Resonators (μHSR). The heat treatment of the samples and its effect on the quality factor (Q) of the resonators is studied. We show that thermal annealing shifts the coefficient of thermal expansion (CTE) of the alloy towards its minimum of ~2ppm/°C, as a result of which the Q of a 29kHz μHSR with diameter of 780 μm increases at least 3 times and reaches 7500 in vacuum.

INTRODUCTION
The widespread success of macro-scale Hemispherical Resonator Gyros (HRG) [1] has been an inspiration and a motivation towards design and implementation of their micro-scale counterparts (μHRG). Although micro-scale freestanding hemispherical shells have been fabricated three decades ago for inertial fusion experiments [2], recent efforts are particularly aimed at their damping and resonant behavior [3,4]. High Q μHSRs can lead to the implementation of high performance resonant gyroscopes that can directly measure angle of rotation instead of angular velocity, or rate of rotation, which most commercial gyroscopes measure today. A range of materials is reported for μHSRs including poly-silicon [3] and silicon dioxide [4], but a potentially high Q metallic μHSR can be advantageous.

Nickel-Iron alloys, a class of metallic materials with exceptional thermal properties are interesting candidates for microelectromechanical systems (MEMS). Elinvar for example, is an Iron-Nickel-Chromium alloy with a zero temperature coefficient of elasticity (TCE). Super-Invar, a less studied variation, contains 64% Fe, 31% Ni, and 5% Co, which provides a CTE very close to zero. Invar36 is a Nickel (36%) Iron (64%) alloy with a very low CTE comparable to that of fused quartz and silica [5]. The small CTE, an anomalous property called the Invar effect [6], can translate into low thermoelastic damping (TED), which makes Invar an attractive material choice for high-Q 3D MEMS resonators. Furthermore, being a conductor gives Invar a significant advantage over other low CTE materials that are mostly insulator as it makes capacitive actuation and sensing possible without requiring additional coating material, which can often reduce Q.

In this work, results from electrodeposition and sputtering of Invar are presented and compared. A process flow for fabrication of Invar μHSRs is introduced; thermal annealing of Invar shells is explored and experimental results related to the effect of annealing on CTE and Q of Invar microshell resonators are provided.

FABRICATION OF INVAR MICROSHELL RESONATORS
Invar μHSRs with capacitive electrodes are successfully fabricated and tested (Figure 1). Two different structural material deposition techniques are examined: electrodeposition and sputtering. Figure 2 demonstrates a sputtered super-Invar μHSR (left), which shows poor structural integrity with visible folding of the shell at the rim, while electroplated Invar microshell (right) shows superior structural integrity compared to sputtered Invar.

In this work, results from electrodeposition and sputtering of Invar are presented and compared. A process flow for fabrication of Invar μHSRs is introduced; thermal annealing of Invar shells is explored and experimental results related to the effect of annealing on CTE and Q of Invar microshell resonators are provided.

Figure 1: An electroplated Invar μHSR with assembled electrodes suitable for actuation and detection of m=2 wineglass modes.

Figure 2: (left) A sputtered super-Invar μshell with poor structural integrity, particularly, the rim is folded towards the center. (right) An electroplated Invar μshell with superior structural integrity compared to sputtered Invar.
on the exposed silicon to form the hemispherical molds (c). A 0.1 µm thick Titanium adhesion layer followed by a 0.5 µm thick Invar or super-Invar as seed layer is sputtered onto the sample. Invar is then electroplated onto the seed layer (d). The surface Invar is polished using a Chemical-Mechanical Planarization (CMP) process leaving invar only in the mold, while the structural Invar shell is protected by a photoresist (PR) layer (e). The structure is finally released using XeF₂ gas which etches the silicon around the shell as well as the exposed Titanium layer (f). On a separate wafer, electrodes are fabricated by etching electrode patterns on a 700µm/2µm/700µm SOI (g). Finally the structure and the electrodes are assembled to form the device (h).

**Electrodeposition of Invar**

Table 1 shows the recipe for electroplating Invar, which is a slightly modified version of what is given in [7]. Particularly, the Ferrous Sulfate concentration is increased from 0.07 M/L to 0.533 M/L. The solution is stirred at 60RPM during the process to maintain uniformity of composition across the sample.

The anode is made of Invar and the cathode houses the sample to be electroplated. The current source provides 15-30 mA/cm² pulsed current (50 ms ON and 30 ms OFF).

Sodium Saccharin is an essential element in the bath to reduce film stress, but should not exceed a limit, above which it will leave carbon residue on the film. Pulsed current is used as a technique to reduce the stress even more, to prevent film delamination, which occurs around the Invar composition.

After each deposition, the composition is verified by Energy-Dispersive X-ray Spectroscopy (EDS) as shown in Figure 4 to confirm 36% Nickel and 64% Iron composition. Using our current plating setup, the electroplated film shows 9% composition variation across the surface, which reduces the fabrication yield.

**HEAT TREATMENT OF INVAR**

Heat treatment of the alloy is needed in addition to accurate composition to reach the Invar effect. The necessity of annealing is sometimes linked to the lattice allotropicity of the atoms. The Invar effect only develops if the material is in γ-phase (Face-Centered Cubic: FCC), and not in α-phase (Body-Centered Cubic: BCC), and heat treatment is needed to transform the BCC units to FCC units [8]. The importance of annealing is also identified in association with the lattice allotropicity.

**Table 1: The electrolyte recipe**

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Sulfamate</td>
<td>0.51 M/L</td>
</tr>
<tr>
<td>NiBr₂</td>
<td>0.04 M/L</td>
</tr>
<tr>
<td>FeSO₄</td>
<td>0.533 M/L</td>
</tr>
<tr>
<td>H₃B0₃</td>
<td>24 g/l</td>
</tr>
<tr>
<td>Sodium Saccharin</td>
<td>4 g/l</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>1 g/l</td>
</tr>
<tr>
<td>Wetting Agent</td>
<td>0.01 g/l</td>
</tr>
<tr>
<td>pH</td>
<td>3.0</td>
</tr>
<tr>
<td>Temperature</td>
<td>40 (°C)</td>
</tr>
<tr>
<td>Current density</td>
<td>20-25 mA/cm²</td>
</tr>
</tbody>
</table>

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with the fact that the CTE is low only for the crystallites of the alloy and not the grain boundaries. Although Invar can be in FCC phase as electroplated, annealing is still needed to enforce grain growth and therefore to reduce the CTE to its minimum [9].

The annealing is performed by placing the samples in an air tight chamber, which is purged for 5 minutes. The temperature is ramped up to the annealing temperature (620 °C) in 300 seconds and held for 5-30 seconds. After the samples are annealed, the temperature is ramped down slowly to ambient.

The electroplated invar exists as pure FCC (γ-phase) directly after deposition, which can be inferred from the graphs of Figure 5. The top graph shows XRD pattern of the electroplated Invar film right after deposition only with peaks corresponding to γ-phase; the left peak with Miller index (1,1,1) and the right peak (2,0,0). The lower graph of Figure 5 shows that the width of γ(1,1,1) peak has decreased after annealing, which based on Scherrer formula translates into bigger grain size [9]. To confirm the XRD results, more accurate measurements of the grain size are done directly from the SEMs shown in Figure 6 with visible grain boundaries. The average grain sizes before and after annealing are measured as ~20 nm and ~70 nm respectively.

Both XRD analysis and direct grain size measurements confirm that the grains of electroplated Invar films, already in FCC phase, grow significantly as a result of annealing. Therefore, the annealed films are expected to show a lower CTE.

**CTE Characterization**

To measure the CTE of electroplated Invar before and after annealing, Vernier microgauge structures [10] are designed and fabricated. As shown in Figure 7, the microfabricated Vernier-like structure measures expansions or contractions of the test beams caused by residual or thermal strain. They bend the slope arm and create an angle, which is magnified by the indicator beam and can be visually observed at the Vernier gauge.

Similar to the μHSR, Invar is electroplated on sputtered titanium and super-Invar layers, and after release some samples are annealed to be compared to non annealed ones. The deflections of the Vernier are measured at two temperatures, namely -20 °C and 50 °C and are compared to calculate the CTE of electroplated Invar before and after annealing using analytic models given in [10]. The average measured CTE of electroplated Invar before annealing is 5.9075 ppm/°C. After annealing at 620°C for 5-6 seconds, the CTE of the film was reduced. The average CTE of annealed Invar was found to be 2.975 ppm/°C. The lowest CTE was observed as 2.09 ppm/°C for an Invar film which was annealed at 620 °C for 6 seconds.

**RESONATOR MEASUREMENT**

Several Electroplated Invar μHSR were fabricated and electrically tested. Devices were capacitively actuated and sensed using low resistivity capacitive silicon electrodes. The dimensions of the tested shells, their measured m=2 frequencies and Q factors are given in table 2. As shown in Figure 8a, an annealed electroplated Invar μHSR with a frequency of 29kHz shows a Q of 7500. Measurements of non-annealed electroplated Invar μHSRs show Qs in the range 1500-3000; an example is shown in Figure 8b. Sputtered super-Invar μHSRs demonstrated a lower Q of ~1300 as shown on Figure 8c.

As the trend shows, annealed Invar μHSRs have the best performance in terms of energy loss. Also, a small frequency split of 27 Hz was measured from an annealed Invar microshell between the two degenerate m=2 modes.

**CONCLUSION**

Electrodeposition and sputtering of Invar36 for fabrication of 3D μHSRs were presented and compared. It was shown that electroplated Invar is a suitable material for fabrication of 3D curved microstructures with high quality factor. The effect of annealing on CTE was studied and was linked to grain size growth after annealing. Experimental data on CTE were provided using microfabricated Vernier-like structures. Thermal annealing shifts the CTE of the
Table 2: Measured $Q$-factors of electroplated Invar µHSRs with different dimensions. The annealed device shows the highest $Q$

<table>
<thead>
<tr>
<th>Annealing</th>
<th>Diameter (µm)</th>
<th>Thickness (µm)</th>
<th>$f_{m=2}$ (kHz)</th>
<th>$Q$ factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>No</td>
<td>910</td>
<td>3.5</td>
<td>11.64</td>
<td>2,000</td>
</tr>
<tr>
<td>No</td>
<td>910</td>
<td>5.2</td>
<td>19.70</td>
<td>1,862</td>
</tr>
<tr>
<td>No</td>
<td>910</td>
<td>5.2</td>
<td>19.70</td>
<td>2,000</td>
</tr>
<tr>
<td>No</td>
<td>910</td>
<td>5.2</td>
<td>18.3</td>
<td>3,196</td>
</tr>
<tr>
<td>No</td>
<td>780</td>
<td>5.6</td>
<td>36.1</td>
<td>1,500</td>
</tr>
<tr>
<td>Yes</td>
<td>780</td>
<td>5.2</td>
<td>29.08</td>
<td>7,567</td>
</tr>
</tbody>
</table>

Figure 8: The $m=2$ frequency response of a non-annealed (a), an annealed (b) electroplated Invar µHSR with thickness of 5.2µm and diameters of 920 µm and 780µm respectively. The $m=2$ frequency response of a sputtered super-Invar shell is shown in (c).

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REFERENCES

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