In Situ Localized Annealing for Contamination Resistance and Enhanced Stability in Nickel Micromechanical Resonators

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ABSTRACT

A technique in which a micromechanical resonator is operated at large amplitudes while *in situ* localized annealed to temperatures exceeding 880°C is shown to be an effective method for both removal of surface contaminants and for possible "redistribution" of the structural material towards substantially higher quality factor Q and greatly enhanced drift stability. The technique not only provides insight identifying contamination as a dominant mechanism for Q-degradation in nickel-plated micromechanical resonators exposed to uncontrolled environments, but also offers a convenient method for restoring a contaminated device to its original high-Q ($Q=14,172$) characteristics.

I. INTRODUCTION

Vibrating micromechanical resonators ("µresonators") with Q's in the tens of thousands and frequencies approaching 100MHz have recently shown great potential for replacing the off-chip high-Q passives used in communication transceivers, with both size and power reduction advantages [1,2]. Thus far, polysilicon has been the material of choice for mid-VHF applications, with Q's on the order of 8,000 in this range [2]. However, as frequencies rise beyond VHF, losses arising from the finite resistivity of doped polysilicon become a concern [1]. For this reason, metals are of great interest as structural materials. Unfortunately, the majority of plated or deposited metals used in micromechanical applications have so far shown relatively low Q's (e.g. only 3,000 in nickel [3]), inadequate thermal stability [4], and unacceptable hysteresis and drift.

This work introduces a post-fabrication procedure that significantly improves the performance of nickel µresonators. In particular, a technique in which a µmechanical resonator is operated at large amplitudes while *in situ* localized annealed to temperatures exceeding 880°C has been shown to substantially enhance quality factor Q and drift stability. After first describing the nickel plating process used to realize resonators in Section II, this paper details the *in situ* localized annealing technique, providing operational specifics and experimental data in Sections III and IV, respectively, then concluding with a discussion of possible mechanisms behind observed enhancements.

II. FABRICATION OF NICKEL µRESONATORS

Figure 1 summarizes the fabrication sequence used to realize nickel microstructures for this work. The process begins with the growth of 2µm thermal oxide on a <100> silicon substrate to serve as an isolation layer for eventual devices. Next, 300Å of titanium (for adhesion), then 2700Å of gold, are evaporated in succession, then patterned via lift-off to form interconnects (Fig. 1(a)). Next, aluminum is evaporated to a thickness of 1.8µm to serve as a sacrificial layer to be removed later. Vias are then patterned into the Al using an $H_3PO_4/HNO_3/H_2O$ wet etchant, exposing the underlying Ti/Au in specific areas (Fig. 1(b)), over which nickel is plated to serve as rigid anchor supports for eventual resonators. The nickel plating is timed to achieve a planarized wafer surface immediately before the start of structural layer processing (Fig. 1(c)).

Structural layer processing begins with a 200Å-thick evaporation of Ni to serve as a seed layer for subsequent Ni plating. A mold is then formed by patterning a 6µm-thick AZ 9260 photoresist to expose areas where structural material is to be plated (Fig. 1(d)). The Ni structural layer is then plated over exposed areas to a thickness of 3µm in a nickel sulfamate solution at 50°C (Fig. 1(e)). Plating is performed via an apparatus in which the wafer (actually, its conductive seed layer) serves as the cathode in a electrochemical
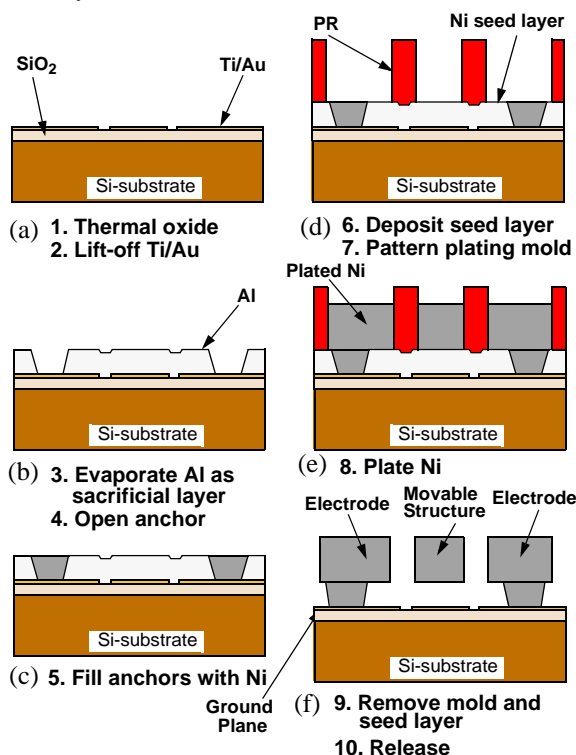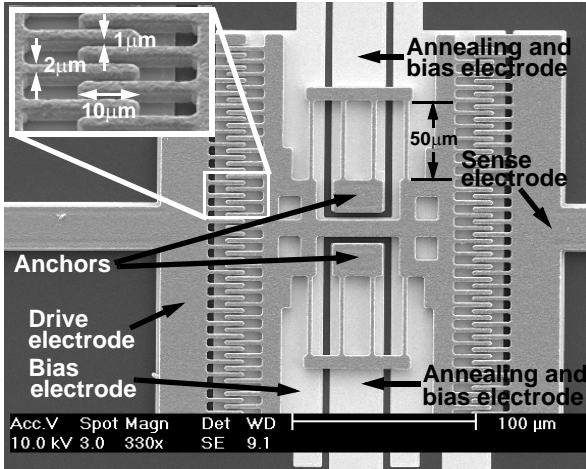

Fig. 1: Nickel surface micromachining process flow.

Fig. 2: SEM of a fabricated Ni comb-driven microme-chanical resonator equipped with annealing pads.
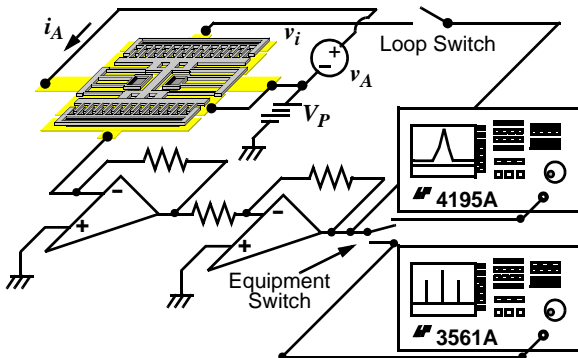

Fig. 3: Schematic showing circuits needed to affect self-sustained oscillation with *in situ* annealing.

reaction, the anode being a Ni rod. The best Ni films were obtained using a cathode-to-anode current density of 2.5mA/cm^2. After plating, the mold and the seed layer are removed using wet chemical processes, and structures are released in a $K_4Fe(CN)_6$/NaOH solution that attacks Al while leaving Ni and Au intact (Fig. 1(f)). Etch selectivities in this etchant are both >100:1 for Al:Ni and Al:Au. Fig. 2 presents the scanning electron micrograph (SEM) of a fabricated μreso-nator with labels for key features and dimensions.

III. *In Situ* LOCALIZED ANNEALING

As detailed previously in [2], localized annealing is instigated by passing a current i_A from anchor to anchor through a given resonator with sufficient magnitude to heat the resonator to a temperature T_A that often exceeds 800°C. Methods for analytically determining the temperature profile over a resonator under specified annealing conditions include both thermal circuit modeling and finite-element modeling (FEM), both previously discussed in [2]. Using the FEM approach, typical T_A's achievable on the Ni μresonator shuttle are 443°C for v_A=0.5V and 886°C for v_A=1V.

This work introduces *in situ* localized annealing, in which the subject device is annealed *while* operating. Figure 3 presents a schematic indicating the bias and excitation scheme required to achieve self-sustained
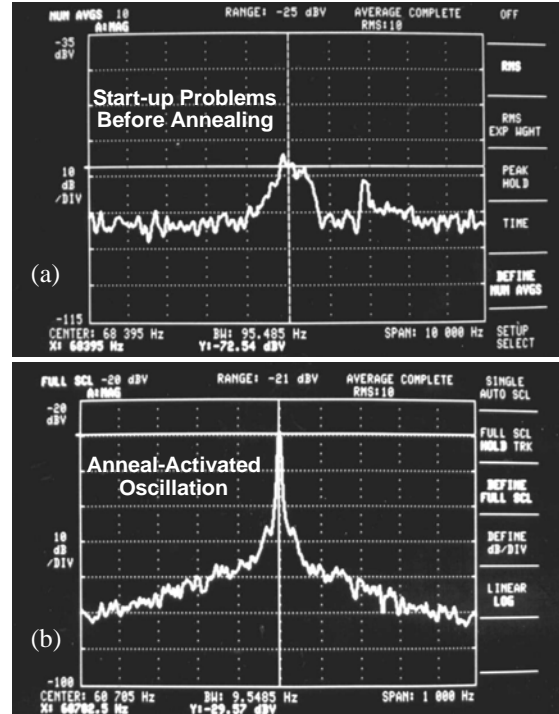

Fig. 4: Measured output spectra for an oscillator refer-enced to a Ni-plated micromechanical resonator (a) before and (b) after *in situ* localized annealing.

oscillation of a μmechanical resonator with *in situ* localized annealing [2]. As shown, an off-chip, two-stage, transresistance amplifier is used to instigate and sustain oscillation, while a dc or pulsed voltage is applied between resonator anchors to both affect annealing and amplify force components at the frequency of the input voltage v_i. As will be seen, the ability to simultaneously operate and anneal a given nickel device via this set-up is instrumental for achieving stable device performance within a reasonable time frame.

IV. EXPERIMENTAL RESULTS

A custom-built vacuum chamber capable of housing a small pc board with on-board electronics under pressures down to 50μTorr was used to test devices. This board was designed to also fit snugly onto an MMR temperature controllable cantilever to allow thermal stabilization of die-level devices. On-board electronics consist essentially of the circuit shown in Fig. 3 with additions to allow switchable selection of either open- or closed-loop operation. Before any measurement is made using this set-up, the board is taken to a high temperature under 50μTorr pressure until outgassing is reduced to an acceptable level.

Upon initial testing, approximately 50% of freshly fabricated Ni μresonators showed inadequate Q and poor drift stability. *In situ* localized annealing as described above was then applied to deficient devices to improve their performance. Q increases obtained via this procedure were immediate, as verified by the oscillator output spectra of Fig. 4 taken before and just seconds after the start of *in situ* annealing. Here, an-
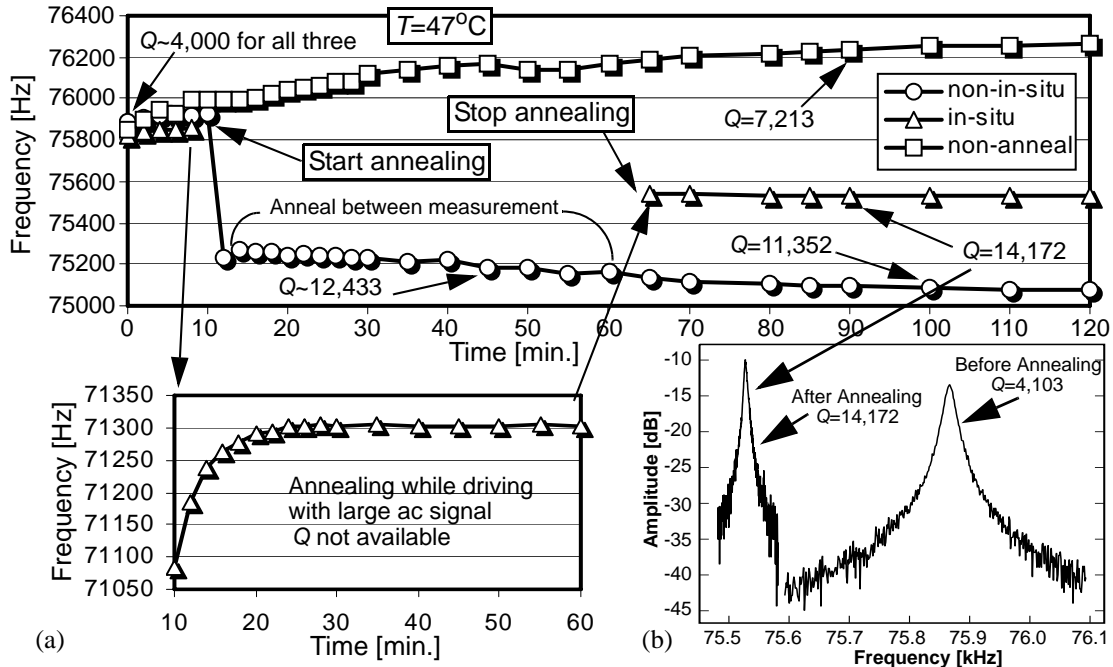
Fig. 5: (a) Frequency vs. time plots comparing various procedures for enhancing the Q and stability of Ni μmechanical resonators. (b) Measured frequency spectra before and after *in situ* anneal-activated post-processing.

nealing clearly instigates start-up by raising resonator Q. Once established, the oscillation peak of Fig. 4 continued to drift, but could be stabilized by *in situ* annealing for a period of about 10 minutes, after which no further frequency drift was observed.

Stability Enhancement.

To better quantify these observations, Fig. 5 presents a plot of frequency versus time for three Ni μresonators, each with Q's around 4,000 immediately after fabrication. Of the three resonators, one is merely operated right after fabrication, while each of the others is subjected to one of the following post-process steps: (1) static annealing, in which the resonator is localized annealed to 886°C while stationary (i.e., not operated simultaneously), and annealing is stopped when spectra are taken; and (2) *in situ* annealing, in which the resonators are operated continuously while localized annealed to 886°C. After one hour, any post processing is stopped, and all resonators are operated and monitored continuously at a well controlled temperature of 47°C. At this point, the *in situ* annealed resonator was clearly the most stable, showing a much smaller frequency drift rate (specifically, no measurable drift in this time period) than any of the others. In addition, Q increases are observed for all resonators, the smallest for the unannealed one (Q=7,213), the largest for the *in situ* annealed one (Q=14,172). This data and the fact that *in situ* annealing achieves the largest gains in both Q and frequency drift stability, suggests the possibility of a defect redistribution/removal mechanism, activated when the structure is operated ("worked") and aided by elevated temperatures. A defect redistribution/removal mechanism is further supported by Fig. 5(b), which shows measured frequency characteristics for a nickel μresonator before

and after *in situ* annealing, showing not only a much higher Q after annealing, but also a reduction in frequency. The observed frequency decrease implies that the structural nickel material has softened—an event often associated with defect redistribution/removal in macroscopic annealing procedures [5].

The length of time required to completely stabilize a given device was consistently found to be about 10 minutes for annealing temperatures on the order of 880°C. The required time increases when the anneal temperature is reduced, giving evidence of a time-temperature dependence, further supporting a redistribution-based mechanism. In addition, although pulsed annealing currents (e.g. to achieve rapid thermal annealing [2]) were able to instantaneously raise Q's, they were unable to stabilize devices as effectively as long term dc anneals. In particular, a square wave v_A signal took longer (in total anneal time) to stabilize a given device than a straight dc anneal.

Environmental Q-Degradation and Restoration.

Once enhanced, μresonator Q's remain high as long as they are kept in a controlled, vacuum environment. When removed from vacuum and exposed to less controlled environments, resonators often again exhibit low Q when returned to vacuum (although not as low as their values right after fabrication), but in most cases could again be taken back to high Q's via another localized annealing step.

To quantify these findings, the effect of humidity on device operation was investigated as follows:
(i) *In situ* anneal a device in vacuum to achieve good stability and high Q;
(ii) place the device in a humidity chamber for a specified length of time and at specific values of percent humidity and temperature;

Table I: Environmental *Q*-Degradation and Anneal-Activated Restoration

No.	Environments	Before Exposure		After Exposure		Annealing Techniques (Time[min])	After Annealing	
		f_o [Hz]	Q	f_o [Hz]	Q		f_o [Hz]	Q
1	50°C / 90% humidity / 90min	79,524	7,695	87,174	3,920	*in situ* (10)	87,176	4,198
2	50°C / 90% humidity / 15min	76,436	7,269	no resonance		static (0.2)	76,501	7,235
3	50°C / 80% humidity / 90min	81,016	6,688	91,476	2,179	*in situ* (10)	91,504	2,600
4	50°C / 80% humidity / 15min	88,392	8,302	88,511	8,475	n/a	n/a	n/a
5	50°C / 70% humidity / 90min	77,101	7,042	78,333	4,061	*in situ* (10)	77,133	7,269
6	50°C / 70% humidity / 60min	77,038	7,081	77,101	7,042	n/a	n/a	n/a
7	on an outside tree / 3 days	72,923	6,706	no resonance		*in situ* (20)	73,031	6,318
8	under a stairway / 3 days	82,768	6,941	no resonance		*in situ* (8)	83,790	7,418
9	inside EECS Building / 3 days	85,679	7,348	85,442	6,417	*in situ* (13)	85,664	7,252

(iii) return the device to vacuum and test immediately without any annealing, determining the frequency, *Q*, and stability, if possible; and

(iv) *in situ* anneal the device and again determine frequency, *Q*, and stability.

The above procedure was applied to several nickel μresonators under a variety of humidities and exposure times, and at chamber temperatures of 27°C and 50°C. Table I presents a subset of the results obtained at 50°C, plus results obtained under less controlled, but more natural, environments. (Note that the *Q*'s in this experiment are lower than in Fig. 5, because the devices used were obtained from a different run.) The major observations over the course of these experiments can be summarized as follows:

(1) Although all devices were designed to be identical, a rather large spread in frequencies is seen (~13%), about 30X higher than observed for polysilicon counterparts.

(2) None of the environmental exposures noticeably affected the drift stability of any of the devices once returned to and operated in vacuum.

(3) Exposures at 27°C had no effect on any devices, even at 90% humidity for 90 min.

(4) Resonators exposed to environments with lower humidity-time totals at 50°C (e.g., cases 4, 6) were unaffected in both *Q* and stability.

(5) For larger humidity-time totals (e.g., cases 1, 2, 3, 5) resonator *Q*'s were degraded, some so severely that they could not be operated after exposure. *In situ* localized annealing was able to recover original *Q*'s in all cases, except 1 and 3.

(6) *Q*'s were recovered instantly by annealing alone (i.e., *in situ* not required).

(7) Under particularly severe conditions (e.g., 1), *in situ* annealing could not recover the original *Q*.

(8) For most cases in Table I, the frequency increases after exposure to humidity.

One possible explanation for observation (8) is mass removal by either (i) corrosion; or (ii) cleaning of device surfaces, both enabled via exposure to humid environments and accelerated by annealing.

Observation (2) seems to preclude a wet contaminant-removal-based mechanism for stability enhance-ment and further supports the previously mentioned mechanism where the combined operation (i.e. "working") and annealing of a nickel μresonator serve to redistribute or eradicate defects from the material towards a more relaxed, stable, and higher *Q* state.

Observation (6) gives evidence that before annealing, the *Q*-degradation in the Ni-plated devices of this work is dominated by surface contamination, which is removed by high temperature annealing to achieve higher *Q*'s. Once contaminants are removed, structural defects or internal stress fields take over as dominant loss mechanisms (best removed via *in situ* annealing, as shown in Fig. 4). In addition, the fact that the *Q* of already annealed devices changes only when subjected to the rather high humidity-time totals of cases 1 and 3 in Table I, but not for others, gives some evidence that localized annealing under controlled environments may even also serve to passivate device surfaces against further contaminant-based *Q*-degradation.

V. CONCLUSIONS

Post-fabrication *in situ* localized annealing procedures have been demonstrated that greatly enhance the stability and *Q* of nickel-plated μmechanical resonators. Defect redistribution/removal and contaminant removal are postulated as likely mechanisms behind observed performance enhancements. The described procedures have been shown to enhance the performance of nickel resonators both immediately after fabrication and even after prolonged exposure to harsh environments. With these procedures, and with process modifications to improve frequency tolerances, nickel and other metals may soon become reasonable candidates for high-*Q*, μmechanical applications, such as communications components or inertial sensors.

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