Solidly mounted resonators aging under harsh environmental conditions

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Abstract. A contribution to reliability studies of Solidly Mounted Resonators (SMR) submitted to harsh environments such as temperature and humidity is presented. Electrical, structural and chemical monitoring of representative parameters is performed by means of RF, DC characterizations and also X-ray diffraction coupled to X-fluorescence to assess aging in microstructures. Results indicate that humidity affects samples stronger than high temperature. From viewpoint of robustness, non-negligible effects of SiO₂ mass-loading on antiresonance and resonance frequencies are reported. Drifts of parameters for a lonely resonator and filter transmission are both in good accordance. Finally, the need of a full sheet passivation layer is demonstrated in order to protect metals and Aluminum Nitride (AlN) against oxidation and pollutant compounds respectively.

1. Introduction
Nowadays, allocated frequency spectrum of emerging wireless telecommunication systems is extending from 500 MHz to 6 GHz [1], and the number of functions provided by those systems (image and audio data processing) is steadily increasing. Integration of such systems is space consuming and requires ultimate miniaturization of the RF active and passive electronic devices. Our work focuses on one kind of Bulk Acoustic Wave (BAW) resonators now widely used to build RF-filters, separating information from crowded spectrum. RF-Filters based on SMR have been lately reported as a suitable alternative to former Surface Acoustic Wave (SAW) resonators, lumped LC elements and dielectric ceramics. BAW technology appears superior to others in term of low size, performances, and offer conceivable on-chip integration [2]. Nevertheless, even if SMR processing is now well controlled, poor literature is still reported about their failure mechanisms after aging under harsh environmental conditions [3,4]. Their abilities to withstand at such aging directly condition their packaging type, either hermetic or not. Cost of encapsulation in MEMS devices is a critical point and necessitates thorough optimization for making RF-filters competitive on the market. In this context, our work is a kind of contribution to reliability assessment. Impacts of humidity and temperature have been carried out on various resonators. Electrical monitoring coupled to structural and chemical analysis gives us precious information about performance degradations.

2. Process design
A schematic presentation of SMR cross section is given in figure 1. The studied devices are composed of a piezoelectric thin film sandwiched between two metallic electrodes, and the bottom one is deposited on acoustic isolation. This one could be realized by air cavity [5], but in the case of SMR, a Bragg reflector prevents acoustic loss into the silicon substrate. Bragg reflector enhances sturdiness for subsequent chip dicing operations compared to Air-Gap type Resonators (AGR). SMR exhibits also better temperature stability and power handling, but suffers from acoustic energy losses in Bragg reflector. In opposite, acoustic energy is well confined in AGR, giving better quality factors. AlN as piezoelectric thin film material is a promising candidate material for filtering applications because of
good electromechanical coupling, high thermal conductivity, chemical compatibility with RF-IC and low temperature process favoring above-IC integration. Bottom and top electrodes are often made of Molybdenum (Mo) following its suitable acoustic impedance. Antiresonance frequency is mainly determined by the thickness of the AlN layer and decreases with increasing thickness. Samples tested in this work include a 1.45 μm-thick AlN film, Mo is used for top and bottom 0.2 μm thick electrode with the latter separated from the substrate by a 20nm-thick titanium or 15 nm-thick AlN thin film. The Bragg reflector is composed of a succession of quarter wave layers (λ/4) of high and low acoustic impedance (W and SiO₂ respectively) for a targeted resonance frequency of 2 GHz. Moreover, some resonators were mass-loaded by a thin SiO₂ layer of about hundred nanometer thick deposited on top electrode only, with the purpose to locally decrease their resonance frequencies (~3% of the carrier frequency) for subsequent ladder (see an example on figure 2) or lattice filter constructions. A last process consists in depositing a Ti-Au thin film (Titanium-Gold) on Mo pads to improve electrical and mechanical contacts with probes. In this early work, wafers are not passivated by silicon nitride, often used as a protection layer.

![Figure 1. Schematic cross section of SMR.](image1)

![Figure 2. Example of 3/2-T-ladder architecture.](image2)

3. Aging procedures and measure uncertainties

Harsh environmental conditions, performed at ambient pressure in our study, are derived from JEDEC standard aging tests, commonly employed in microelectronic area. Principally, such tests in case of SMR would favor element intrusion in presence of humidity, metals oxidation at elevated temperature and delamination of layers during quick deviations of temperature. Typically, humidity consists in 85°C/85% RH, temperature storage was 250 °C, just below temperature process (300°C) and temperature cycling ranged from −65 °C up to +150 °C with 22 minutes dwelling time and less than one minutes for transition from low to high temperature. Drifts of many resonators and several filters responses were monitored during aging by removing samples from ovens, and then by probing wafers with a vector network analyzer HP8720ES. In addition to RF devices, DC patterns as Van Der Pawn and Kelvin contact allowed to measure electrical resistivity of Ti-Au, Mo and Ti-Au/Mo contact resistances respectively, before and during temperature storage.

Before aging assessment, the first experiments consisted in evaluating measure uncertainties. Such procedure aims to dissociate impact of harsh environments from systematic errors in scattering-parameter measurements. Confidence interval is determined at 95 %. In worst case, antiresonance frequency (f_a) uncertainties don’t exceed +/-305 KHz; +/-400 KHz for resonance frequency (f_r); +/-0.058 % for coupling factor (k_r); +/-16 and +/-21 for antiresonance (Q_a) and resonance (Q_r) quality factors respectively. Uncertainties for the two resonance frequencies are very weak and represent less than 0.02 % of the carrier frequency. Coupling factor (determined from both resonance frequencies) is also accurate but results about quality factors highlight the need of a very good frequency resolution. For instance, near 5.5 % of error on quality factors is computed for resolution as well as 500 KHz.
Complementarily to electrical characterizations, X-ray diffraction was used to monitor AlN’s texture evolution or Mo degradation, and X fluorescence for examining chemical properties [6]. In RF-MEMS area, complexity of multi-layer architecture has to be taken into account. With at least 4 different thin layers presenting various mechanical properties, RF-MEMS devices require new characterization technique developments. Several methods have been already developed for thin layer characterization using X-ray [7] or in-situ wafer curvature measurement [8]. Moreover, some combination with Finite Elements modeling is often necessary for an accurate and quantitative stress determination [9]. In the present work we have developed a new setup using simultaneously the three X-ray capabilities for complementary nanoscale characterizations: X-ray diffraction for micro structural analysis (structure, texture and stress measurement), X-ray tomography for morphological imaging and X-ray fluorescence (XRF) for chemical analysis [10]. A micro focus X-ray beam was used for local scanning across the resonator.

4. Results
At least 24 miscellaneous samples are heated up to 250 °C for 1000 hours cumulated times, figure 3 shows typical frequencies evolutions in respect to aging time for bare resonators and mass-loaded ones. It appears clearly that mass-loaded resonators remain quasi-invariable even at 1000 hours while the two resonance frequencies decrease of about several megahertz for bare ones.

![Graph 3](image1.png)

**Figure 3.** Drifts of "fi" and "fa" in respect to virgin state of resonators having different active areas, with and without SiO₂ mass-loading vs. 250°C storage time.

![Graph 4](image2.png)

**Figure 4.** Drifts of "Qr" in respect to virgin state of resonators having different active areas and shapes (apodized (1,2)) vs. 250°C storage time.

Regarding quality factors, Qₐ vary randomly and no information is extracted. However, shift of Qₐ is significant for all resonators even if drift amounts occur close to uncertainties, as depicted figure 4. Qₐ diminishing can be correlated to electrical DC measures performed on the various metals constitutive stack, see in table 1. Resistivity and contact resistance of all conductors have increased after temperature storage, certainly due to oxidation of Mo and diffusion of Au inside Ti (more resistive). As a result, resistance of the electrical path between acoustic area and signal supplier increases. This phenomenon can be easily verified by modifying access resistance in the Butterworth Van Dyke model [11]. A slight increase of this resistance reduces quality factor at resonance frequency only. Regarding band pass filters built in ladder configuration with four shunt and four series resonators, previous effects observed on mass-loaded (shunt) resonators compared to their bared counterparts (serie) let us expect shrinkage of bandwidth mainly by right side, related to bare resonators. Figure 5
shows $S_{21}$ transmission of filters for 1000 hours duration and confirms results observed on resonators with an 11.5 MHz shrinking inside bandwidth.

**Table 1.** Evolution of electrical resistivity and contact resistance of metals constitutive stack, before and after 660 hours of temperature storage (250°C).

| Resistivity ($\mu$Ohms.cm) | Contact resistance (Ohms) |
|----------------------------|---------------------------|
| Mo Bottom                  | Ti-Au/Mo Bottom           |
| Virgin 11                  | 0.09                      |
| Aged 12                    | 0.32                      |
| Mo Top                     | Ti-Au/Mo Top              |
| 11                         | 4.4                       |
| 12.5                       | 47.9                      |

From viewpoint of thermal investigations and considering uncertainties, temperature cycling (−65°C to +150 °C), even after 1000 hours, do not affect significantly resonance frequencies, coupling factor and quality factors. Thermal shocks of JEDEC standards would be more appropriate to age resonators.

![Figure 5. Impact of a thermal storage (1000 hours, 250°C) on a filter response.](image1)

![Figure 6. Drifts of "fr" and "fa" in respect to virgin state of resonators having different active areas, with and without SiO$_2$ mass-loading vs. humid storage time (85°C/85% RH).](image2)

The most severe alteration was pointed out after humid storage. Monitoring of resonators in humidity was stopped at 240 hours, because no responses occurred in most of them at 660 hours. No more time is required to conclude that humidity damages easily resonators and filters. Nevertheless, SiO$_2$ mass-loading still seems to act as a protection layer on top electrode, as illustrated figure 6. It protects resonators up to 240 hours regarding the two resonance frequencies. For non mass-loaded ones, drifts increase up to 72 hours duration and decrease afterwards to reach few megahertz decades below virgin state. Therefore, measures reveal that overall trend of coupling factors tend to lower in respect to aging time, about more than 0.6 % after 72 hours and even 1 % in some cases. Tendency of the two quality factors are also to decrease more than 100 from their original values. Figure 7 illustrates typical aging of most resonators. Resonator behaviors are well correlated to the filter ones as shown figure 8. Bandwidth and insertion loss increase up to 72 hours and decreases afterwards until failures. It is noteworthy that mass-loaded resonators are less but also severely damaged at 660 hours.

In the three analyses, different sizes and shapes of top electrodes are found to have no particular effects on whole reliability results (Temperature, Humidity).
Figure 7. Typical impact of humidity storage (85°C/85% RH) on resonator's impedance before and for cumulated durations of 72 and 240 hours.

Figure 8. Impact of humidity storage (85°C/85% RH) on a filter response before and for cumulated durations of 72 and 660 hours.

We try to correlate electrical and X-ray measures. On a virgin device, the AlN shows a <0002> fiber texture with a Full Half Width Maximum (FWHM) less than 2°. The Molybdenum also exhibits a strong <110> fiber texture. After humid storage, one can observe a change on the micro structural grain of AlN layer. The fiber texture decreased while the FWHM of the rocking curve increased. The results presented on figure 9 confirm the change of the AlN grain microstructure as a consequence of the humid storage treatment.

Figure 9. Rocking curve of the AlN layer before and after aging under humid atmosphere.

Figure 10. GIXRF analysis performed on Si reference and before/after humid storage.

A highly sensitive grazing angle incidence chemical analysis (GIXRF) was also performed [8]. For example, we were able to detect an involuntary pollution with chlorine species induced by the chemistry during the fabrication process, dicing or oven contamination. One can notice the presence of a chlorine peak in the spectrum related to the accidental pollution, as shown in figure 10. This pollutant is highly suspected to be the origin or AlN's texture degradation since chlorine compounds could be used for AlN's etching. Moreover, decrease of coupling factor, previously mentioned, is the result of FWHM enlarging in rocking curve [12]. It means that grain's orientation decay along polar
axis due to a preferential chemical corrosion of the AIN layer with respect to the crystallographic orientation.

5. Conclusion
Following the perspective of reliability study on this new RF-MEMS, impacts of harsh environments are investigated. In one hand, it reveals that AIN's texture decay along polar axis in humid atmosphere. In consequence, coupling factor decreases and chlorine compounds are highly suspected to be the origin of degradation. In the other hand, high temperature storage tends to facilitate metal oxidation reducing quality factor at resonance.

Furthermore, in term of resonance and antiresonance frequencies, SiO2 mass-loading acts as a local passivation layer and protect resonators compared to their bare counterparts. Filter transmissions are in good accordance with results obtained on lonely SMR. It is concluded that such aging is unbearable in view to keep guard-band specifications of filters, which are very strict between transmission and reception path [13]. In order to guarantee good operation of the system, a real passivation layer is necessary to protect all parts of resonators. On-going researches are now focused on effects of a silicon nitride passivation layer deposited on full sheet that let us expect less degradation. We also ought to evaluate robustness under thermal shock, electrostatic discharge (ESD) and eventually mechanical shocks, vibrations, accelerations.

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