<http://www.diva-portal.org>

Postprint

This is the accepted version of a paper published in *IEEE Sensors Journal*. This paper has been peer-reviewed but does not include the final publisher proof-corrections or journal pagination.

Citation for the original published paper (version of record):

Sturesson, P., Zahra, K., Lena, K., Greger, T. (2017)
Ceramic Pressure Sensor for High Temperatures: Investigation of the Effect of
Metallization on Read Range
IEEE Sensors Journal, 17(8)

Access to the published version may require subscription.

N.B. When citing this work, cite the original published paper.

Permanent link to this version:

<http://urn.kb.se/resolve?urn=urn:nbn:se:fhs:diva-7342>

Ceramic Pressure Sensor for High Temperatures – Investigation of the Effect of Metallization on Read Range

Peter Sturesson^{1,2,3}, Zahra Khaji², Lena Klintberg² and Greger Thornell^{1,2}

¹*Ångström Space Technology Centre, Dept. of Engineering Sciences,
Uppsala University, Uppsala, Sweden*

²*Division of Microsystems Technology, Dept. of Engineering Sciences,
Uppsala University, Uppsala, Sweden*

³*Dept. of Military Sciences, Swedish Defence University, Stockholm, Sweden*

Corresponding Author: peter.sturesson@angstrom.uu.se

Abstract— A first realization of membranes by draping a graphite insert with ceramic green body sheets, and a study on the relationship between circuit metallization, made by double-layer screen-printing of platinum and electroplating of silver on top of platinum, and the practical read range of ceramic LC resonators for high-temperature pressure measurements, are presented. As a quality factor reference, two-port microstrip meander devices were positively evaluated. To study interdiffusion between silver and platinum, test samples were annealed at 500, 700, and 900 °C for 4, 36, 72, and 96 hours. The LC resonators were fabricated with both metallization methods, and the practical read range at room temperature was evaluated. Pressure sensitive membranes were characterized for pressures up to 2.5 bar at room temperature, 500 and up to 900°C. Samples electroplated with silver exhibited performance equal to or better than double-layer platinum samples for up to 60 hours at 500°C, 20 hours at 700°C, and for 1 hour at 900°C, which was correlated with the degree of interdiffusion as determined from cross-sectional analysis. The LC resonator samples with double-layer platinum exhibited a read range of 61 mm, and the samples with platinum and silver exhibited a read range of 59 mm. The lowest sheet resistance, and, thereby, the highest read range of 86 mm, was obtained with a silver electroplated LC resonator sample after 36 hours of annealing at 500°C.

Index Terms—pressure sensors, ceramic, high temperatures, wireless reading, LC resonator, annealing, quality factor, read range

I. INTRODUCTION

Wireless sensors for harsh environments in general, and those based on resonator components in particular, have experienced a renaissance in recent years. Harsh environments, comprising high temperatures and pressures, high power throughput, radiation and corrosive substances, are of great interest for many enterprises, including military

development activities, as demonstrated in DARPA's HERMiT program [1]. Combustion engines, such as fighter jet engines, are being developed to exert gas at temperatures close to 2000 °C, [2], and an explosion from a 100 kg trinitrotoluene (TNT) load, generates an over-pressure zone ranging from 1658 to 2.7 bar 1 and 15 m from its center, respectively [3]. Challenges for space vehicles have been addressed for a long time, [4], where the pressure in launch boosters with liquid propellants can reach about 7 bar and the temperature can exceed 1000 °C. Also, future near-sun missions, that require hardware able to sustain at least 850 °C, are being planned and studied [5-6].

For devices required to sustain temperatures of 400°C and above, ceramic materials have been suggested, and High- and Low-Temperature Co-fired Ceramics (HTCC and LTCC respectively), described in [7-8], are now well-established technologies. After first having been presented and demonstrated with LTCC technology for temperatures up to 400 °C [8], LC resonator circuits for wireless pressure reading have been further developed [9]. From 2013, Xiong et al. and Tan et al. have presented several versions from both LTCC and HTCC with demonstrations of pressure measurements at temperatures up to 800°C [10-12]. In parallel with this, cavity resonators working at the same temperature have been developed and presented [13]. In 2015, a thermomechanical study on LC resonators fabricated from HTCC, including a demonstration of wireless reading of pressures up to 2.5 bar at 1000°C, was published [14].

The use of magnetically coupled devices implies a restriction in read range between the active reader antenna and the passive LC resonator. The devices presented in [10] and [11] operate at 25 and 28 mm, respectively, and in [13], a reading distance of 10 mm is reported.

As a representation of device efficiency, and thereby also read range, the quality factor is often used. For LC resonators, it is mainly derived from resistance, capacitance and

inductance. For high-temperature environments, the resistance, being temperature dependent, can be assumed to influence the quality factor. In [10-12, 14], capacitor plates on membranes provide a pressure-dependent capacitance, implying a function-based influence of the quality factor. As the geometry of the conducting patterns is relatively stable, mainly governed by the thermal expansion coefficient (CTE) of the substrate material, changes in quality factor due to elevated temperatures are determined by changes in dielectric constant and resistivity.

The most common ways to design resonators and their read-out system in order to achieve high read ranges are presented in [15]. However, alternative readout principles have been suggested [16-17]. Also, an alternative component design with a larger distance between an inductive communicator and a capacitive sensor node, in order to increase the total distance between sensor node and reader antenna, have been presented [11].

The fabrication of homogeneous and geometrically stable pressure sensitive membranes from HTCC has also proved to be a challenge [10]. Commonly, gas cavities are fabricated by milling, laser cutting and embossing of ceramic sheets in green body state [18]. The membrane then entails another ceramic sheet, suspended over the cavity. Fugitive materials are usually used to prevent cavities from collapsing during lamination and firing. In [19], several methods to fabricate controlled cavities in ceramic green tapes have been presented. In some of these, no mechanical structuring was made. Instead, enclosed fugitive material inserts, acting as mechanical mandrels, internally embossed channels and cavities.

This paper presents a study of the effect the circuit metallization have on the practical read range for ceramic LC resonators with pressure sensitive membranes fabricated with the simplified HTCC process of internal embossing, in part resembling that of making ravioli. Moreover, since electromagnetically coupled devices have short read ranges, and their feasibility of being applied as remote sensing nodes in harsh environments therefor can be challenged, an attempt to improve the read range of LC resonators by decreasing the resistance in their coils by two metallization schemes: platinum screen printed in two layers, and silver electroplated on a single platinum layer, is made.

Microstrip devices are utilized as indicators of the effect on the device's quality factor from these metallizations. Resistance test samples are fabricated to study the interdiffusion between silver and platinum with time and temperature. Furthermore, the relationship between coil resistance of LC resonators and their practical read range is studied.

II. THEORY

The resonance frequency, f , of LC resonator circuits is governed by their capacitance, C , and inductance, L , and is described by:

$$f = \frac{1}{2\pi\sqrt{LC}}. \quad (1)$$

For parallel plate capacitors, the capacitance is described by:

$$C = \epsilon_0 \epsilon_r \frac{A}{d}, \quad (2)$$

where ϵ_r is the relative permittivity for the dielectric material between the capacitor plates, ϵ_0 is the permittivity of vacuum, A is the plate area, and d is the distance between the plates.

The inductance can be described by the modified Wheeler equation:

$$L = K_1 \mu_0 \frac{n^2 d_{avg}}{1 + K_2 r}, \quad (3)$$

where K_1 and K_2 are design-related constants with the respective values of 2.34 and 2.75 for square coils, μ_0 is the permeability of free space, n is the number of coil-turns, d_{avg} is the mean value of the outer and inner coil diameter, and r is the fill ratio defined as (outer length - inner length)/(outer length + inner length) [20]. Here length is referring to the side of the coil.

The quality factor, Q , of series LC resonators is described by:

$$Q = \frac{1}{R} \sqrt{\frac{L}{C}}, \quad (4)$$

where R is the conductor resistance. Combining (1) and (4), the quality factor can be expressed as [21]:

$$Q = \frac{2\pi f L}{R}, \quad (5)$$

The coil resistance is affected by the frequency as current crowding can occur, and follows:

$$R(f) = R_{DC} \left(1 + \frac{1}{10} \left(\frac{f}{f_{crit}} \right) \right), \quad (6)$$

where R_{DC} is the DC resistance, f the resonance frequency of the device, and f_{crit} the critical frequency where current crowding begins to have a large influence [22]. R_{DC} is given by:

$$R_{DC} = \frac{\rho l_{tot}}{l_w l_t}, \quad (7)$$

where ρ is the conductor resistivity, l_{tot} the length of the coil, and l_w and l_t are the coil conductor width and thickness, respectively [23]. The critical frequency, f_{crit} , is described by:

$$f_{crit} = \frac{2\pi \cdot 3.1 \cdot \rho(l_w + l_p)}{\mu_0 l_w^2 l_t}, \quad (8)$$

where l_p is the coil conductor pitch [23].

The two layers of metals used here, are treated as two parallel resistors, with resistances R_a and R_b , respectively. In the case of interdiffusion between the two, a boundary layer where the metals form an alloy, acting as a third parallel resistor, is expected. With R_{eff} being the resistance of this layer, the resulting resistance, R_p , can be expressed as:

$$\frac{1}{R_p} = \left(\frac{1}{R_a} + \frac{1}{R_b} + \frac{1}{R_{eff}} \right). \quad (9)$$

According to Nordheim's rule, the effective resistivity, ρ_{eff} , of an alloy can be approximated by:

$$\rho_{eff} = \alpha \rho_a + \beta \rho_b \quad (10)$$

where alpha and beta are the atomic fractions, and ρ_a and ρ_b are the resistivity of the respective metals [24]. For metal layers with identical dimensions, resistivity can be substituted with resistance and (10) can be inserted into (9), and written as:

$$R_p = \frac{1}{\left(\frac{1}{R_a} + \frac{1}{R_b} + \frac{1}{\alpha R_a + \beta R_b} \right)}, \quad (11)$$

A screen-printed 12 μm thick single layer of platinum has a sheet resistance of about 70 $\text{m}\Omega/\text{sq}$ according to initial measurements, and $60.1 \pm 19.8 \text{ m}\Omega/\text{sq}$ according to [6]. Silver, having a resistivity of 15 % of that of platinum [23], is here assumed to have a sheet resistance of 11 $\text{m}\Omega/\text{sq}$ for a single layer of the same thickness. The resulting resistance should then be about 9.1 $\text{m}\Omega/\text{sq}$. If an interdiffused, alloyed layer of equal proportions of silver and platinum, making up a third of the conductor's total thickness, is present, the resistance, according to (11), is expected to increase to about 12 $\text{m}\Omega/\text{sq}$. If the entire conductor has alloyed, the resistance should reach its maximum value, here about 40 $\text{m}\Omega/\text{sq}$.

III. MATERIALS AND METHODS

To understand the correlation between different metallizations and the resulting sheet resistance and read range of LC resonators, the circuit quality factor as a function of metallization is studied. Then the influence of time and temperature on the metal systems and their resistances are investigated. Furthermore, the coil resistance and passive signal return of LC resonators with pressure sensitive membranes are measured and compared for the different metal systems, and the practical read range is evaluated. Finally, the high-temperature performance of LC resonators with both the suggested metallizations is demonstrated.

This study comprises the fabrication of three different structures with the HTCC process: microstrip meander

resonators, resistance test samples, and LC resonators. Below, the manufacturing is first described in general, and then detailed for each structure.

A. HTCC Processing

All samples were manufactured with HTCC technology, utilizing two different platinum pastes (5571/5574-A, ESL, USA). ESL 5571 is a high-resistivity paste with alumina grains mixed with it, whereas ESL 5574-A is a nearly pure, low-resistivity paste [7]. Both pastes were screen-printed on 100 or 150 μm thick ceramic green body sheets (44007-100/150, ESL, USA). Alignment holes were drilled using a PCB plotter (S100, LDKF, Germany) with 1.1-mm diameter spiral drills. All sheets necessary for components were stacked, aligned using a fixture with alignment pins, and packed in a vacuum bag. The bag was then placed in a water-filled in-house built stainless steel chamber, connected to an HPLC pump (HPLC 515, Waters, USA). After being heated to a steady-state temperature of 70 $^\circ\text{C}$, the chamber was pressurized to 200 bar, if nothing else is stated. After this, the components were contoured manually with a cutting blade.

The components were then placed between plates of porous alumina in a high-temperature furnace (ECF 20/18, Entech, Sweden), and heated, fired and sintered as described in detail in [14]. During this, organic binders and solvents in the pastes are removed.

B. Membrane Fabrication

For membranes, suspended over cavities, 8-mm diameter fugitive inserts from graphite paste (4441, ESL, USA) were screen printed in two layers on a 150 μm thick ceramic green body sheet, each layer followed by 15 minutes drying at 50 $^\circ\text{C}$. A second 150 μm thick green body sheet enclosed the finished inserts. The stacks were then stepwise laminated at 70 $^\circ\text{C}$ for 5 minutes at 10, 20, 35 and 50 bar, respectively, and finally for 10 minutes at 70 bar, making the graphite inserts function as form-shaping mandrels, as presented in [19]. Finally the stacks were fired and sintered according to the process described above. Since graphite is the main content of the ESL 4441-paste, this is removed entirely.

C. Additional Metallization

To reduce the resistance of the LC resonators and increase their quality factor and, potentially, their read range, in comparison with those of [14], two methods were employed: screen printing of two layers of ESL 5574-A paste on top of each other, and electroplating of silver on ESL 5571 platinum. (Detaching from the substrate, the ESL 5574-A was found incompatible with electroplating, and therefore excluded from this step.)

Screen printing of two layers was accomplished by drying the first print at 50 $^\circ\text{C}$ for 15 min, and using the pattern alignment feature of the screen printing equipment to print the second layer on top of the first one.

For electroplating, a 200- μm diameter insulated copper wire, connected to the cathode, was attached to the conductive platinum pattern using conductive epoxy (CW2400, Chemtronics, USA). The sample surface was

Fig. 1. Microstrip device in top view (left), and in exploded view (right), displaying its ceramic and metallic layers.

electrochemically cleaned at 5 V for 5 minutes (ActiClean, Spa Plating, UK), followed by silver plating for 40 min (Silver tank plating solution, Spa Plating, UK), using a plating factor of 5 mA/cm^2 and aiming for a silver film thickness of $12 \text{ }\mu\text{m}$, which corresponds to the typical thickness of a screen printed conductor line.

In addition to these double-metallized components, samples with single layers of ESL 5574-A, such as those presented in [14], and ESL 5571, were prepared as references.

D. Microstrip Resonators

For an initial evaluation of the various metallizations, microstrip meander resonators with 2-port connections were manufactured. Having coupling gaps between the feedline and the microstrip, these allow separation of the feedline from the resonator. Exploiting the transmission type of measurement by measuring the S_{21} parameter, from which the loaded quality factor can be calculated, contact resistance influence on measurements and calculations is reduced.

These resonators were designed to have a resonance frequency of 50 MHz, similar to the frequencies of the LC resonators reported in [14], table 1. The meander shape, chosen to decrease the sample footprint size, distanced by $100 \text{ }\mu\text{m}$ to its feeding lines, was screen printed on a green body sheet (ESL 44007-150). A ground plane was screen printed on another sheet, as a bottom layer, and four additional alumina sheets were placed in between these screen printed sheets, Fig. 1. Each sheet had a 90° rotation relative its neighbor sheets in order to avoid stress accumulation between them. Two samples were screen printed with single and double layers of the ESL 5574-A paste, respectively. A third sample was screen printed with the ESL 5571 paste. After lamination, in this case made by a hydraulic laminator (SMP 210, Bungard, Germany) due to the stack size, the samples were contoured to a size of $115 \times 23 \text{ mm}$ and sintered. Each sample was connected with two SMA connectors to a vector

network analyzer, VNA (Fieldfox 9923, Agilent, USA). With this, the forward voltage gain factor (S_{21} parameter) was read and the 3 dB-cut loaded quality factor was measured. Subsequently, the sample with ESL 5571 was silver electroplated, again aiming for a thickness of $12 \text{ }\mu\text{m}$, and re-measured.

TABLE 1
Design parameters for the microstrip devices.

Parameter	Value	Device
Strip length	600 mm	All
Number of turns	25	All
Meander turn radius	3 mm	All
Relative permittivity	10	All
Gap size	$100 \text{ }\mu\text{m}$	All
Conductor width	$900 \text{ }\mu\text{m}$	All
Sample thickness	$700 \text{ }\mu\text{m}$	All
Conductor thickness	$12 \text{ }\mu\text{m}$	5571
	$12 + 12 \text{ }\mu\text{m}$	5571 + Ag
	$12 \text{ }\mu\text{m}$	5574, 1 layer
	$24 \text{ }\mu\text{m}$	5574, 2 layers

E. Temperature Sustainability of the Metallizations

In high-temperature environments, interdiffusion between materials can be expected. For metals, this implies resistivity changes according to (10). To study this, a total of $14 \times 10 \times 3 \times 0.7 \text{ mm}$ resistance test samples were fabricated from 6 layers of $150 \text{ }\mu\text{m}$ thick green tapes (ESL 44007-150). Of these, 12 samples were metallized with the ESL 5571 paste followed by a $12 \text{ }\mu\text{m}$ thick electroplated layer of silver, whereas for references, one sample was printed with two layers of ESL 5574-A paste, and the last one with a single layer of ESL 5571.

After pre-annealing measurement of their sheet resistance, using a four-point probe (CMT-SR2000N, AIT, South Korea), the samples were placed in a muffle furnace (Thermolyne,

Thermo Scientific, USA) and heated to temperatures of 500, 700 and 900 °C in 600, 800 and 1000 s, respectively. For each temperature, four electroplated samples were placed in the furnace and annealed for 4, 36, 72 and 96 hours, respectively. The reference samples were placed in the furnace during the 500°C and 900°C tests for 96 hours each. On completion, the sheet resistance of all samples was sampled four times.

After this, the samples were encapsulated in epoxy and prepared for cross-sectional microscopy. Sputtered with a 400 Å thick layer of gold-palladium, each sample was analyzed with respect to the distribution of platinum and silver, using scanning electron microscopy (LEO 440, Zeiss, UK) and Energy Dispersive Spectroscopy, EDS (EDAX Microanalysis, EDAX, USA).

F. Coil Resistance and LC Resonator Read Range

For measurement of the read range and pressure sensitivity of LC resonators, the circuit design from [14], described in table 2 and displayed in Fig. 2, but with the metallizations introduced here, was used, table 2.

TABLE 2
Design parameters for the LC resonator devices.

Parameter	Value	Metallization
Number of turns	8	All
Conductor width	250 μm	All
Coil turn pitch	500 μm	All
Capacitor area	16 mm^2	All
Capacitor plate distance	250 μm	All
Conductor thickness	15 μm	5574, 1 layer
	15 + 12 μm	5571, w. Ag
	25 μm	5574, 2 layers

Fig.2. Exploded view of the LC resonator design from [14] with the screen printed graphite insert between the ceramic two sheets that the circuit pattern is printed on.

From (1)-(3) & (8), a resonance frequency of 53 MHz, and a critical frequency of about 1100 MHz are predicted for this design. For $\rho = 10.92 \text{ m}\Omega\text{cm}$ [25], (7) gives a R_{DC} of about 14 Ω , and this substituted into (6), gives a contribution of about 0.067 Ω to the DC resistance, implying that the DC resistance is a dominating factor for the frequencies in this work, and, hence, that the devices will not be heavily influenced by current crowding.

Four samples of each metallization were fabricated, table 3. The inductor coil with the top capacitor plate, as well as the second capacitor plate, were screen printed with platinum paste (ESL 5574-A or 5571) on 150 μm thick ceramic green tape sheets (ESL 44007-150). A graphite mandrel, centered on the capacitor plates, was screen printed on the sheet with the second capacitor plate, Fig. 2. The sheets were stacked, and the samples contoured, before the samples, four at a time, were stepwise laminated, as described above. Finally, they were fired.

TABLE 3
Metallizations of LC resonator samples.

Sample name	Pt-paste	No. of Layers
LC5571-1	5571	1
LC5571-2	5571	1
LC5571-3	5571	1
LC5571-4	5571	1
LC5571-1-Ag	5571	2
LC5571-2-Ag	5571	2
LC5571-3-Ag	5571	2
LC5571-4-Ag	5571	2
LC55741-1	5574-A	1
LC55741-2	5574-A	1
LC55741-3	5574-A	1
LC55741-4	5574-A	1
LC55742-1	5574-A	2
LC55742-2	5574-A	2
LC55742-3	5574-A	2
LC55742-4	5574-A	2

As in [14], the samples were powered and read from a 90-mm diameter tunable loop antenna with a matching loop, connected to the VNA. The reader antenna resonance frequency was controlled by sweeping the voltage of a varactor diode (BB182, NXP, The Netherlands), between 4 and 7 V in steps of 0.1 V. As the resonance frequency was swept over a frequency range of 10 MHz in 14 steps, the passive signal return from the sensor devices was monitored, Fig. 3. The maximum of the envelope of the minimas of the 14 S_{11} parameter scans was taken as the resonance frequency. The practical read range was defined as the maximum distance for which this resonance frequency could be resolved.

The coil resistance, sample resonance frequency, and the power return profile from the devices to the reader antenna were measured at room temperature for distances between 0 and up to 118.4 mm in steps of 3.2 mm. Afterwards, samples LC5571-1/2/3/4 were electroplated with silver, and their read range re-evaluated.

Fig. 3. S_{11} sweeps of the reader antenna yielding minimas, the maximum of whose envelope corresponds to the device's resonance frequency. At too low signal return, i.e., too long reading distance, the signal is detectable but no local maximum can be distinguished.

For correlation with the time- and temperature-dependent resistance of the resistance test samples, and for comparison of changes in coil resistance and the read range, sample LC5571-2 was heated in the muffle furnace, first at 500 °C for 36 h, and then at 900 °C for 36 and 72 h, and characterized after each annealing step.

G. High-Temperature Behavior of LC Resonators

One at a time, samples LC55742-2 and LC5571-3, were placed in a custom-made quartz tube furnace, fused in one end and interfaced with a metal flange in the other. The samples were connected to a K-type thermocouple. The tube, visualized in [14], was filled with fine-grained alumina sand for thermal conduction and mass, pressurized with air through the metal flange, and heated from the outside with an oxygen-acetylene flame from 25 to 900 °C in steps of 100 °C. At room temperature, 500 °C and 900 °C, the pressure was increased from 1 bar to 2.5 bar in steps of 0.3 bar. The reader antenna, described above, was placed around the tube in the same plane as the sample, and the resonance frequency measured versus temperature and pressure.

IV. RESULTS

All samples were successfully manufactured. The metal patterns, in Fig. 4, were all intact and exhibited good edge definition. The membranes were bulging slightly upwards from the sample surfaces, but retained an overall flat shape, resembling that of ravioli.

Fig. 4. Samples LC55741-1 (top), LC5571-4 (left) and LC55742-3 (right) after sintering. The slightly bulging membrane is easiest to see on sample LC55741-1.

A. Quality Factor

The loaded quality factor obtained from the S_{21} parameter of the microstrip resonator devices for different metal systems is presented in Fig. 5. For the sample with high-resistivity ESL 5571 paste, and a resonance frequency of 58.7 MHz, the quality factor was about 8 before electroplating, and about 33 afterwards. The low-resistivity ESL 5574-A paste samples exhibited quality factors of 20 for one layer and 40 for two layers of screen-printed metal. The corresponding resonance frequencies were 59.7 and 55.6 MHz.

Fig. 5. Quality factor obtained from the S_{21} parameter measurements on differently metallized microstrip resonators.

B. Temperature Sustainability of Metallization

Fig. 6 presents the sheet resistance of the electroplated test samples as a function of annealing time and temperature. At 500 °C, the mean sheet resistance decreases from 12.9 to 5.7 mΩ /sq. for the first 36 hours, and then increases to 55 mΩ/sq. after 96 hours. At 700 °C, the mean resistivity initially

Fig. 7. Silver and platinum concentration profiles, obtained with EDS, over cross-sections of silver electroplated on ESL 5571 platinum after various degrees of annealing. All profiles start at the top metal surface and end at the interface to the ceramic, as indicated in the first column.

decreases from 19.9 to 15.8 mΩ/sq., and then increases to 46.5 mΩ/sq. after 72 hours, after which it decreases to 40.1 mΩ/sq. after 96 hours. At 900 °C, however, the mean resistance increases immediately and reaches 82.5 mΩ/sq. after 36 hours, where after it decreases to 68.8 mΩ/sq. after 96 hours.

Fig. 6. Sheet resistance of silver electroplated on platinum after various degrees of annealing. The mean value of the reference samples from ESL 5571 and ESL 5574-A are displayed as dashed lines.

C. Time and Temperature Dependency of Silver-Platinum Diffusion

Fig. 7 shows the distribution of platinum and silver over the cross-sections of the resistance test samples as a function of annealing time and temperature.

For the first 36 hours at 500 °C, the metals are still well separated with only a small fraction of silver having diffused into platinum. After 96 hours, there are regions with significant presence of both silver and platinum. At 700 °C, the metal layers are still rather intact after 4 hours, and then start to interdiffuse until they seemingly reach steady state after 96 hours. At 900 °C, this has occurred already before the first analysis.

D. Coil Resistance and Read Range

The mean value and standard deviation of the coil resistance of all LC resonators for all metal systems are given in Fig. 8. For ESL 5571 the mean resistance is 46.3 Ω and 16.1 Ω before and after electroplating, respectively. For ESL 5574-A the resistance is 21.7 and 11.5 Ω for single and double layers, respectively.

Fig. 8. Resistance of the LC resonator coils for the respective metal systems, with bars showing the standard deviation, being, from left to right: 1.7, 0.3, 0.6, and 0.4 Ω .

A comparison of the DC resistance for sample LC5571-2, after electroplating, and the sheet resistances presented in Fig. 8, at 36 h at 500 $^{\circ}\text{C}$ and 900 $^{\circ}\text{C}$, and at 72 h at 900 $^{\circ}\text{C}$, is presented in Fig. 9. The coil resistance was initially 16.5 Ω , as compared with 43.9 Ω before electroplating, and then decreased to 4.8 Ω after 36 hours at 500 $^{\circ}\text{C}$. After 36 hours at 900 $^{\circ}\text{C}$, the resistance was 60 Ω and the coil was semi-detached from the substrate. After 72 hours at 900 $^{\circ}\text{C}$, the resistance was 53.5 Ω , and the coil was almost completely detached from the substrate.

Fig. 9. Comparison between the mean values of the sheet resistance measured for the silver electroplated resistance test samples (black bars and y-axis scale) and the resistance over the coil for sample LC5571-2 after being silver electroplated and annealed (grey bars and y-axis scale).

E. Wireless Signal Return and Practical Read Range

For the LC resonator samples, the mean resonance frequency was 49.9 MHz with a standard deviation of 0.95 MHz. For most of them, the maximum signal return value is almost constant within a short range of about 5 mm and then decreases nearly linearly before leveling out, Fig. 10. Samples LC5571-1 to 4 exhibited an average maximum signal return of about 1.18 dB before they were electroplated and 1.73 dB after. Samples 55741-1-4 and 55742-1-4 exhibited an average maximum signal return of 1.63 dB and 1.74 dB, respectively. The vertical lines display the average practical read range, and at which signal return level this is reached, for each metal system at room temperature.

Fig. 10. Signal return from all passive sensor samples vs. reading distance. The vertical lines mark the average practical read range for each metal system and the error bars represents the standard deviations.

Sample series LC55742 and LC5571-Ag, exhibit almost indistinguishable signal return trends albeit different read ranges, up to 61 and 59 mm at about 0.19 and 0.21 dB signal returns, respectively. Sample series LC5571 and LC55741 (with single layers of the low- and high-resistivity platinum, respectively) exhibit practical read ranges of about 33 and 51 mm at about 0.85 and 0.25 dB signal return, respectively.

Fig.11 displays the signal return behavior for sample LC5571-2 at the different stages of fabrication and annealing. The signal return had reached a maximum of about 2 dB after the sample had been heated at 500 $^{\circ}\text{C}$ for 36 hours, which also resulted in a practical read range of 86 mm.

Fig. 11. Signal return vs. reading distance for sample LC5571-2 before and after silver electroplating, and after annealing at 500 °C for 36 hours. Vertical lines represent the read range for each series.

Fig. 12. Sample LC5571-2 after annealing for 72 hours at 900 °C. The top capacitor plate and large parts of the coil has detached from the substrate surface.

After 36 hours at 900 °C, the read range could not be measured as the coil had detached from the alumina substrate and deformed severely, Fig. 12.

F. High-Temperature Behavior of LC Resonators

The frequency shift with ambient pressure was generally quite linear, yielding sensitivities of 7000, 8100 and 900 ppm/bar at room temperature, 500 °C and 900 °C, respectively, for sample LC55742-2, and 7230 and 23660 ppm/bar at room temperature and 500 °C, respectively, for sample 5571-Ag-2, Fig. 13.

Fig. 13. The pressure dependency of the resonance frequencies of samples LC5542-2 and LC5571-3 at room and elevated temperature.

As for the temperature sensitivity, it differed much, also qualitatively, for these samples, Fig. 14.

Fig. 14. Frequency shift with temperature of samples LC5542-2 and LC5571-3.

V. DISCUSSION

Calculating the quality factor of the LC resonators from the mean coil resistances of the different metal systems, using (1)-(3) and (5), results in quality factors similar to those obtained experimentally from the microstrip devices, Fig. 15. This implies that they may provide a valid representation for LC resonators, at the same resonance frequency, in terms of quality factor, especially in the case of all-platinum metallizations.

Fig. 15. Comparison between loaded quality factors obtained from the S_{21} parameter measurements on the microstrip devices (black), and the quality factor calculated from the coil resistances and geometry of the LC resonators (grey).

The temperature dependency of the resistance of the different metallizations appeared only partly to be consistent with theoretical predictions, and so for different temperatures. As expected, the resistance of the platinum reference resistance test samples was preserved, the reason, of course, being that the sintering temperature is much higher than the annealing temperature. The resistances of the silver electroplated samples exhibited a number of varying behaviors on annealing. The decrease in resistance during the initial 36 hour at 500 °C, Fig. 6, is believed to be caused by surface diffusion of silver which results in a smoother layer with lower resistance. In parallel with this, silver and platinum diffuse into each other, Fig. 7. After 36 hours at 500 °C, this becomes pronounced, and the resistance increases, Fig. 6. The same behavior, although faster, was observed at 700 °C. At 900 °C, this is believed to have occurred fast enough to escape observation completely, given that the first analysis was made after four hours. The diffusion equilibrium, observed after 96 hours at 700 °C and 36 hours at 900 °C, Fig. 7, indicates that platinum and silver have alloyed completely, i.e., throughout the metal thickness. At 500 and 700 °C, the same (equilibrium) phase region should eventually be reached, [26]. At 900 °C, however, a different equilibrium region is reached. As previously calculated using (11), the resistance should be at its maximum when the two metals are fully alloyed. The decrease in resistance that is observed after 72 hours at 700 °C and after 36 hours at 900 °C, when the metals have been fully interdiffused, is believed to be due to recrystallization. The recrystallization temperature of platinum is in the range of 400 to 800 °C [27], and typically at about 500 °C [28].

The extensive alloying may explain why the coil of LC5571-2 detached after 36 hours at 900 °C as the CTE of the alloy is likely different from that of pure ESL 5571 platinum, thus causing stress in the interface between substrate and

conductor.

In conclusion, the behavior presented in Fig. 6 suggests a time and temperature range for when silver-platinum conductors are more favorable, in terms of read range, than platinum conductors. Here, it is implicated that silver electroplated devices would outperform those of double-layered platinum for nearly 60 hours, after some annealing at 500 °C. Annealing at 700 and 900 °C, however, would reduce this time to about 20 hours and one hour, respectively.

Recalling that the signal return behavior was practically indistinguishable between sample series LC55742 and LC5571-Ag, Fig. 10, the difference in read range is explained by the small difference in sheet resistance and, thereby, the quality factor. A higher quality factor makes it easier to resolve the maximum from the envelope of the S_{11} minimas of the reader antenna, also if the level of signal return is maintained. It also explains why the practical read ranges are reached at higher signal return levels the lower the sheet resistance and quality factor is. Thus, the reading method, explained in chapter 3.5 and visualized in Fig. 3, is somewhat favorable for low coil resistances.

The time and temperature behavior of the sheet resistance, as presented in Fig. 6, could be reproduced with sample LC5571-2 for resistance and read range after 36 hours at 500 °C, and, at least, for resistance after 72 hours at 900 °C. The relationship between practical read range and coil resistance can be visualized by plotting the resistance values obtained from the different metal systems of the LC resonator coils versus the measured practical read ranges, Fig. 16.

Fig. 16. Relationship between practical read range and average coil resistance of the LC resonator coil for different metal systems.

The temperature dependent behavior of the relative frequency shift of sample LC55742-2 at room temperature is similar to what has been observed previously [10-12, 14], taken into consideration the difference of membrane dimensions and their resulting pressure-induced deflection, as

described in [29]. At 500 °C, the pressure sensitivity of the same sample, Fig. 13, is somewhat higher, which is contradictory to previously observed behavior.

For sample 5571-3-Ag, the frequency increases with temperature up to 700 °C, where it suddenly drops, Fig. 14. Here bimorphic behavior, due to the difference in CTE between silver and platinum, can contribute to a curvature of the membrane. As silver has a CTE of about 19 and platinum one of about 9, [30], thermal expansion will make the membrane bulge outwards from its cavity. This would increase the capacitor plate distance and also the resonance frequency. At 700 °C, however, the completed silver and platinum interdiffusion will cease the bimorphic behavior and allow the membrane to assume another steady-state shape, maybe accompanied by a transition similar to bistable buckling.

The pressure sensitivity at 900 °C was too low to be extracted with good certainty, but at 500 °C the sensitivity was higher than at room temperature, Fig. 13, and, to the authors' knowledge, in fact larger than for any previously reported device.

Minimizing the thickness of the platinum layer and coating it with a layer of silver thick enough to saturate it with large margin, may solve the non-favorable time and temperature behavior. This would also improve the overall performance of the LC sensor node. Electroplating is advantageous as it follows the original platinum pattern without needing a mask. But plated films are often subjected to residual stress, which puts a limitation to the thickness. This is particularly important when the adhesion to the substrate is poor. Also, whereas the platinum pastes are thermomechanically matched to alumina, silver is not, wherefore an increased amount of silver will affect the device's performance over temperature, even if the metal layers *per se* have reached steady state. It should also be noted that excessive plating would decrease the isolation distance between the coil turns and, hence, change the inductance.

Finally, obtaining long read ranges with inductive coupling is challenged by the characteristics of the magnetic field induced from the reader antenna, as the field strength decreases cubically with increasing distance. Thus, from 100 mm, which is about the maximum read range presented in this study, to 1000 mm, it reduces with three orders of magnitude [31].

VI. CONCLUSIONS

- Both two-layer platinum and silver on platinum metallizations were successfully realized.
- The practical read range is improved by both metallizations, with the silver electroplated LC resonators performing in class with those of double-layered high-resistivity platinum.
- Fabricating membranes by draping ceramic green sheets over a sacrificial insert, has, for the first time, been successfully demonstrated.
- Microstrip resonators have shown to indicate the quality factor of LC resonators well.
- The performance of silver electroplated sensors is improved by heating at 500 °C for up to 36 hours.
- Silver electroplated sensors can sustain performance for up to 60 hours at 500 °C, 20 hours at 700 °C and about 1 hour at 900 °C.
- Interdiffusion of silver and platinum is deemed the main reason for changes of sensor performance with time and temperature.
- The quality factor is indicative of the practical read range for samples with similar passive signal return.

ACKNOWLEDGMENT

The authors like to thank Leif Kuzavas at the Microstructure Laboratory, Uppsala University, for valuable advice, and the Ångström Workshop for experimental facilities. Martin Berglund and Anders Persson at the Ångström Space Technology Centre are acknowledged for valuable input. The Knut and Alice Wallenberg foundation is acknowledged for laboratory facilities.

REFERENCES

- [1] C. T. C. Nguyen, (2012, June). “*The Harsh Environment Robust Micromechanical Technology (HERMiT) Program: Success and some unfinished Business*”, Presented at Microwave Symposium Digest (MTT), [Online]. Available: <http://ieeexplore.ieee.org/document/6259750/>
- [2] W. C. Wilson and G. M. Atkinson, “Passive Wireless Sensor Applications for NASA’s Extreme Aeronautical Environments”, *IEEE Sensors*, vol. 14, no. 11, pp. 3745-3753, 2014.
- [3] Ngo, T., Mendis, P., Gupta, A. and Ramsay, J., 2007. “Blast loading and blast effects on structures—an overview.” *Electronic Journal of Structural Engineering*, vol. 7, pp.76-91.
- [4] F. L. Bagby, “Materials in Space” in “*Advances in Space Science*” 1 Ed. Academic Press, 1960, ch. 2, pp. 177.
- [5] Y. Guo, J. McAdams, M. Ozimek and W. Shyong (2014, May). “Solar Probe Plus Mission Design Overview and Mission Profile”, in *24th International Symposium on Space Flight Dynamics*, [Online], Available: http://issfd.org/ISSFD_2014/ISSFD24_Paper_S6-2_Guo.pdf
- [6] G.A. Landis and P.C. Schmitz, (2008, July) “Solar Power System Design for the Solar Probe+ Mission”, in *6th International Energy Conversion Engineering Conference (IECEC)*, [Online], Available: https://aldryn-media-eu.s3-eu-central-1.amazonaws.com/icarus-devel-16327/live/public-media/project_docs/Power_Supply.pdf
- [7] M. Berglund, A. Persson, and G. Thornell, “Evaluation of dielectric properties of HTCC alumina for realization of plasma sources,” *J. of Electronic Materials*, vol. 44, No. 10, pp. 3654–3660, 2015.
- [8] M.A. Fonseca, J.M. English, M. von Arx, and M.G. Allen, “A Wireless Micromachined Ceramic Pressure Sensor for High-temperature Applications”, *IEEE J. of MEMS*, vol. 11, No. 4, pp 337-343, 2002.
- [9] G.J. Radosavljevic, L. D. Zivanov, W. Smetana, A.M. Maric, M. Unger, and L.F. Nad, “A Wireless Embedded Resonant Pressure Sensor Fabricated in the Standard LTCC Technology”, *IEEE Sensors Journal*, vol.9, No. 12, pp.1956-1962, 2009
- [10] J. Xiong, S. Zheng, Y. Hong, J. Li, Y. Wang, W. Wang, and Q. Tan, “Measurement of Wireless Pressure Sensors Fabricated in High-Temperature Co-fired Ceramic MEMS Technology”, *J. Zhejiang University-SCIENCE C (Computers & Electronics)*, vol. 14, pp.258-263, 2013.
- [11] Q. Tan, H. Kang, J. Xiong, L. Qin, W. Zhang, C. Li, L. Ding, X. Zhang, and M. Yang, “A wireless passive pressure microsensor fabricated in HTCC MEMS technology for harsh environments,” *Sensors (Basel)*, vol. 13, no. 8, pp. 9896–9908, 2013.
- [12] Q. Tan, C.Li, J. Xiong, P. Jia, W.Zhang, J. Liu, C. Xue, Y. Hong, Z. Ren and T. Luo, “A High Temperature Capacitive Pressure Sensor Based on

Alumina Ceramic for in Situ Measurement at 600 °C”, *Sensors*, Vol. 14, pp. 2417-2430, 2014.

- [13] H. Cheng, G. Shao, S. Ebadi, X. Ren, K. Harris, J. Liu, C. Xu, L. An and X. Gong, “Evanescence-mode-resonator-based and Antenna-integrated Wireless Passive Pressure Sensors for Harsh-environment Applications”, *Sensor and Actuators A: Physical*, vol. 220, pp. 22-33, 2014.
- [14] P. Sturesson, Z. Khaji, S. Knaust, L. Klintberg and G. Thornell, “Thermomechanical properties and Performance of Ceramic Resonators for Wireless Pressure Reading at High Temperatures”, *J. of Micromechanics and Microengineering*, vol. 25, No. 9, 2015.
- [15] R. Nopper, R. Has and L. Reindl, “A Wireless Sensor Readout System – Circuit Concept, Simulation, and Accuracy”, *Trans. On Instrumentation and Measurement*, vol. 60, no. 8, pp. 2976-2983, 2011.
- [16] H. Zhang, Y. Hong, B. Ge, T. Liang and J. Xiong, “A Novel Readout System for Wireless Passive Pressure Sensors”, *Photonic Sensors*, Vol. 4, No. 1, pp. 70-76, 2014
- [17] H. Zhang, Y. Hong, T. Liang, H. Zhang, Q. Tan, C. Xue, J. Liu, W. Zhang, and J. Xiong, “Phase Interrogation Used for a Wireless Passive Pressure Sensor in an 800 °C High Temperature Environment”, *Sensors*, Vol. 15, No. 2, pp. 2548-2564, 2015.
- [18] L. E. Khoong, Y.M. Tan and Y.C. Lam, “Overview on Fabrication of Three-Dimensional Structures in Multi-layer Ceramic Substrate”, *J. of European Ceramic Society*, Vol. 30, pp. 1973-1987.
- [19] Method of using sacrificial materials for fabricating internal cavities in laminated dielectric structures, by K. A. Peterson. (2009, February 24). *Patent US7494557 B1*. [Online]. Available: Google Patents.
- [20] S.S. Mohan, M. del Mar Hershenson, S.P. Boyd and T.H. Lee, “Simple Accurate Expressions for Planar Spiral Inductances”, *IEEE J. of Solid-State Circuits*, vol. 34, no 10, pp. 1419-1424, 1999.
- [21] H. J. De Los Santos, “Elements of RF Circuit Design” in “*RF MEMS Circuit Design*”, 1st Ed., Artech House, 2002, ch. 2.2.4, pp. 36.
- [22] W. Kuhn and N. Ibrahim, “Analysis of current crowding effects in multilayer spiral inductors”, *IEEE Trans. Microw. Theory Tech.*, vol. 49, no. 1, pp. 31 – 8, 2001.
- [23] M. A. Fonseca 2007 “Polymer/ceramic wireless MEMS pressure sensors for harsh environments: High temperature and biomedical applications”, Ph.D. dissertation, Sch. of El. And Comp. Eng. Institute of Technology, Atlanta, Georgia, 2007, pp. 102.
- [24] W. Gao and N.M. Sammes, “Classical Theory of Electrical Conduction and Conducting Materials” in “*An Introduction to Electronic and Ionic Materials*”, 1st Ed. World Scientific Press, 1999, ch. 2, sec. 2.7.2, pp. 25. M.J Woodman, “The Thermal Conductivity and Electrical Resistivity of Platinum”, *Platinum Metals Review*, vol. 10, no. 4, pp. 132-132(1), 1966.
- [25] P. Durussel and P. Feschotte, “A Revision of the Binary System Ag-Pt”, *J. of Alloys and Compounds*, Volume 239, pp. 226-230, 1996.
- [26] Y. N. Loginov, A. V. Yermakov, L. G. Grohovskaya and G. I. Studenok, “Annealing Characteristics and Strain Resistance of 99.93 wt.% Platinum”, *Platinum Metals Review*, Vol. 51, No. 4, pp. 178-184, 2007.
- [27] F. C. Campbell, “Recovery, Recrystallization and Grain Growth” in “*Elements of Metallurgy and Engineering Alloys*”, 1st, Ed, ASM International, 2008, ch. 8.2.1, pp. 125.
- [28] M. Di Giovanni “Thermal Effects on Physical Properties of Materials” in “*Flat and Corrugated Diaphragm Design Handbook*”, M Dekker INC, ch. 12, pp. 135-136, 1982.
- [29] F. Cverna (2002, Jan), Thermal properties of Metals, ASM International, Materials Park, Ohio. [Online]. Available: <http://www.owlnet.rice.edu/~msci301/ThermalExpansion.pdf>
- [30] K. Finkenzeller, “Physical Principles of RFID Systems” in “*RFID Handbook*”, 3rd Ed., John Wiley & Sons Ltd, 2010, ch. 4, pp. 64.

Peter Sturesson (M’08) was born in 1981 in Storuman, Lappland, Sweden. He received a M.Sc. degree in engineering materials in 2008 from Uppsala University, Sweden. He is currently pursuing a PhD in microsystems engineering at the Ångström Space Technology Centre, Uppsala University and at the Swedish Defence University.

From 2008 to 2012 he was an Aeronautical Engineer in the Swedish Armed Forces, receiving the rank of Captain in 2010 and a position as Development Officer at the Air Warfare Centre in Uppsala, Sweden. His current research is within the field of microsensors for harsh environments.

Zahra Khaji was born in 1983 in Tehran. She received her Bachelor degree in electronics engineering from Iran University of Science and Technology in 2006 and her M.Sc. degree in design and implementation of ICT products and systems from Swedish Royal Institute of Technology (KTH) in 2009. She is currently working as a PhD student at the Division of Microsystems Technology at

Uppsala University, researching microsensors for very high temperatures.

Lena Klintberg was born in 1972. She received her M.Sc. in Engineering Physics from Uppsala University, Sweden 1997, and her PhD in Material Science from the same university in 2002. She is now working as a researcher at the Division of Microsystems Technology at Uppsala University, focusing microsensors that can tolerate high temperatures and high pressures.

Greger Thornell received his PhD in Materials Science in 1999, and now holds the position of professor in Engineering Physics at the Dept. of Engineering Sciences, Uppsala University, Sweden. Thornell has, for several years, also been working as VP Operations, and CTO in the electronics and frequency control industry.

Currently, however, his role as the director of the Ångström Space Technology Centre at Uppsala University gets most of his time. His main professional interest is microsystems technology for harsh environments..