Micromachined Ceramic-based Chipless LC Resonator for High-Temperature Wireless Sensing Application in Harsh Environments

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Abstract: The primary objective of this work was the fabrication and testing of a wireless LC resonator based on micro-patterned electroceramic materials for the monitoring of high-temperature systems. The two-dimensional planar LC resonator sensors were designed and simulated using ANSYS Maxwell software, and these sensors were then fabricated from electrically conductive $\text{La}_2\text{NiO}_4/\text{Al}_2\text{O}_3$ particulate inks. The patterning and deposition of the ink were completed using a novel micro-casting process onto Al_2O_3 ceramic substrates, and the final pattern was bonded onto the substrate at 1200°C for 2 h. The final patterned materials were characterized using X-ray diffraction (XRD), scanning electron microscope (SEM), and four-point conductivity to characterize the phase and microstructure

development, and the resultant electrical conductivity (at 500-1200°C), respectively. The frequency shift with respect to temperature was measured, which is directly related to changes in the sensor's dielectric permittivity and pattern dimensions. The sensors were characterized at 500 – 1000°C in an ambient atmosphere with an RF signal ranging from 10 – 80 MHz at 175 kHz·s⁻¹ sweep rate. A new robust and adaptive signal processing approach was introduced to increase the degree of freedom for analyzing the wireless sensors.

1. Introduction

Wireless communication is very desirable in high temperature applications as it allows sensors to be placed at critical sensing locations where making electrical connections to the sensor is difficult and degradation of these electrical lines may cause altered sensor response due to connection and interconnect changes. The major limitation with the conventional temperature measurements using solid-state sensors such as thermocouples, resistance temperature devices (RTD), and thermistors is that one end of the sensor filament should reside in a relatively cold zone. These devices cannot be applied in a sophisticated high-temperature system due to several design incapacities and the interconnect issues as discussed above. The passive feature of the sensors is critically important as the extremely harsh environment renders the use of batteries for power to be impractical or infeasible. To mitigate the issue of high temperature sensing in harsh environments for sophisticated systems, a passive wireless temperature measurement strategy has been implemented by exploiting various wireless communication technologies. Among various reports, recent studies show that the surface acoustic wave (SAW) and RF-based wireless resonator mechanisms can be applied to high temperature sensor application since these two mechanisms do not require an external power source for the operation [1-7]. R. Behanan utilized the SAW mechanism coupled with an RF antenna for various mechatronic application in ambient environmental condition [3]. Unfortunately, many of the high-performance piezoelectric materials

and the metal electrical connections (and electrodes) used in the fabrication of SAW sensor are limited for these harsh conditions. However, RF-based sensors do not require multi-functional materials and metal electrodes, where the properties may change drastically when exposed to the high temperatures and mixed-atmospheric conditions. RF-based sensors can be fabricated from conductive and stable ceramic materials, where materials with more predictable intrinsic properties (with temperature) can be utilized, and the sensor may be further protected by a barrier coating. In addition, these sensors can be fabricated with no lead wires that extends through the high temperature system, which means that the lead wire influence on the sensor measurements is not present.

The conventional RF sensors are made up of a LC resonator with an integrated antenna [8-11]. RF powered inductor/capacitor (LC) technology shows great promise to be embedded within (or on) active components. In most cases, these LC resonators are printed or deposited on the surface of a component to form a planar structure composed of an inductor (L) and an interdigitated capacitor (IDC) connected in series. The fundamental mechanism of the sensor is based on change in capacitance with respect to the temperature [12, 13]. The change in capacitance will alter the resonant frequency which is unique to every temperature. The shifts in the resonant frequency can be wireless detected and temperature data can be inferred. The inductor which is connected in series to complete the LC resonator circuit is used to absorb and re-radiate the electromagnetic radiation which enables wireless interrogation capability of the sensor.

Fig. 1. Schematic representation of passive wireless passive temperature sensor setup.

A RF signal is transmitted to the LC sensor by an interrogator antenna as shown in the fig. 1. As the interrogator antenna radiates the RF signal, it excites the LC sensor through mutual inductance. The LC sensor reradiates the RF signal back to the interrogator antenna. The strongest components of the reradiated RF signal are at the LC sensors resonant frequency. The resonant frequency of the LC sensor is determined by the LC circuit. The resonant frequency of a LC circuit can be written as,

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

where, f_r is the resonant frequency, L is the inductance, and C is the capacitance of the sensor. The capacitance is inversely proportional to the resonant frequency which shows, as the capacitance increases, the resonant frequency will decrease. In general, the dielectric permittivity of most dielectric ceramic materials, such as alumina and zirconia, increase with temperature [8-12]. As the temperature of these electroceramic material increases, the resonant frequency of the LC resonator will decrease or in other words, the peak will shift towards the left side of the spectrum.

The IDC and capacitor may be deposited onto stable dielectric substrates such as alumina (Al_2O_3) or yttria stabilized zirconia (YSZ). The “fingers” of the IDC and the spiral inductor are usually fabricated using a conductive material. As discussed above, the change in capacitance as a function of temperature can be attributed to: 1) a change in dielectric permittivity of substrate, 2) the thermal elastic strain causing a change in dimension of the IDC/inductor pattern, and 3) a change in the conductivity of the IDC/inductor pattern. Among these factors, the former of the two has a well-established relation to the change in resonant frequency as a function of temperature. Although, electrical conductivity plays a role in the overall sensor response, there are no reports showing its effect on the sensor response. In general, higher conductivity is preferred for an effective change in the resonant frequency. Theoretically, a minimal change in electrical

conductivity will not affect the sensor response because the other two mechanisms dominates over the electrical conductivity. When the electrical conductivity is lower, there will be a significant shift in the resonant frequency because lower conductive material leads to lower capacitance as well as mutual inductance.

LC sensors were shown previously in literature to be used as passive wireless sensors at high temperatures, but stability issues with the conductive material still needs to be addressed. The sensors reported in the literature are predominantly fabricated by depositing precious metals such as silver or platinum on ceramic substrate by thick film technology [8-14]. For example, the sensor presented by E. Birdsell was fabricated from platinum (Pt) onto a ceramic substrate [6]. Unfortunately, the LC components were quite large (>3 inch in minimum dimension) and were not able to fit onto a singular ceramic substrate. The IDC was also found to be unstable and was not operated for many cycles, since Pt (and other precious metals) are known to de-wet and form hill-lock structures during high-temperature operation when deposited onto oxide substrates. Also, there is a large difference in coefficient of thermal expansion between Pt and the oxide substrates, which will cause thermomechanical issues. Although, metals may provide better signal response in the low to moderate temperature regime, they cannot be applicable in a high temperature regime; therefore, the use of a highly conductive ceramic may be a feasible alternative. As with the E. Birdsell work, the dimensions of the reported sensors are large (again, >3 inch in minimum dimension) to integrate with sophisticated systems, such a fuel cell, gasifier, turbine blades, and other micro high temperature systems [6]. An alternate fabrication approach is needed in order to miniaturize the dimension of the sensors to overcome the geometric constraints of the current fabrication approach [8-14].

In this work, a pure ceramic and electroceramic-based LC resonator (sensor) was fabricated, which is capable of measuring temperatures to 1000°C and above. The sensor was fabricated with

an electrically conductive electroceramic composite patterned on a polycrystalline alumina (Al_2O_3) substrate. We introduced a novel fabrication process for the miniaturization of the sensor without compromising the expected wireless signal response. The miniaturization technology discussed in this work allows for the integration of a passive wireless sensor to numerous applications to monitor the temperature and health of the high-temperature system.

In this paper, we also introduce a novel approach for processing the received wireless response from the temperature sensor. The conventional approach is to track changes in the resonant frequency with the expectation that the inductance and/or capacitance of the sensor will change with temperature in a predictable way [8-14]. This requires that the electrical characteristics of the substrate and electrode materials and their interactions can be well modeled as temperatures vary, and that the material properties are stable over many heating and cooling cycles. In practical scenarios, material properties are not known accurately, and they may change (particularly for the wired interconnects) during heating and cooling cycles. To mitigate these issues, we have chosen to measure the response of the sensor over a wide band of frequency around the expected resonant frequency (which may not be precisely correct). In order to achieve this goal, we created a database of known signature wideband frequency responses at each temperature of interest. This permits the measurement of unknown temperatures by comparing the measured wideband frequency response with the database of known responses. Thus, the temperature measurement problem is transformed into a signal matching problem, which is a well-studied fundamental issue in statistical digital signal processing. Applications in biometrics, RADAR ranging, digital communications, and other areas use a similar method to analyze and correlate measured signals to databases of known signals. This approach has the advantage of not requiring the tracking of any particular resonant frequency. All that is required to measure temperature accurately is that the wideband frequency response changes in some measurable way as the temperature changes. Our

approach can even be made adaptive, i.e., it works when material properties change over time simply by creating new signatures at desired time intervals.

2. Experimental section

2.1. Modeling and simulation of the LC resonator

ANSYS Maxwell (Ansys Inc., Canonsburg, PA) software was used to perform initial modeling and simulation of the LC resonator. ANSYS Maxwell is a low frequency electromagnetic (EM) field simulator. The software utilizes finite element analysis to simulate the inductance, mutual inductance, capacitance, EM fields, and other key features of the 2D or 3D models of the sensors. The software allows one to select the materials used in the 2D and 3D models. The electrical properties of the materials (electroceramic composites and the dielectric substrate) were entered into the software for the computational analysis. ANSYS Maxwell is also able to build nonlinear equivalent circuits from the 2D and 3D models and perform frequency analysis on these circuits. Fig. 2 shows the schematic of the 2D representation of the modelled passive wireless sensor for high temperature application.

Fig. 2. Schematic representation of modelled (a) square planar inductor, (b) interdigitated capacitor (IDC) and (c) LC resonator circuit.

For the square planar inductor, we used a fifteen-turn (n) inductor coil. The coil was 0.15 mm wide with 0.15 mm spacing between turns. The outer diameter was 19 mm with an inner diameter of 10.3 mm, as shown in table 1. An interdigitated capacitor (IDC) was chosen for this work, since this type of capacitor could be easily patterned or deposited onto planar surfaces and substrates. An

IDC is specified by the following physical characteristics: number of fingers (n), the width of the fingers (w), the spacing between the fingers (G), and the distance between the fingers (λ). A target resonant frequency of 13.56 MHz was chosen, and this was the basis for choosing a suitable capacitance which resulted in the target frequency whose specifications are listed in table 1. Using these physical characteristics, a 3D model of the square planar inductor and IDC were built in ANSYS Maxwell and its inductance and capacitance were simulated. The inductance and capacitance values are estimated as 5.25 μ H and 25.5 pF for the physical specifications shown earlier.

Table 1. Specifications used in this work for the square planar inductor and IDC (all units are in mm, except n which is number of turns or fingers).

Electrical Component	w	n	d_{out}	d_{in}	s	λ	G	β	α
Inductor	0.15	15	19	10.3	0.15	-	-	-	-
IDC	0.2	80	-	-	-	0.8	0.1	0.1	20

2.2. Digital signal processing methods for temperature measurement

As discussed above, our signal processing approach differs from the state-of-the-art in that we do not track a single resonant frequency. Instead, we measure the wideband frequency response of the sensor around the expected resonant frequency to capture frequency changes that cannot be modeled or expected. This includes resonant frequency changes, but our approach will work even if the resonant frequency does not change as expected, as long as the wideband frequency response changes in any measurable way as temperature varies. Once we have the wideband frequency response signal (a vector), we compare it to a database of frequency response vectors at known temperatures, which we call signatures. This is similar to matched filtering, which is used in RADAR systems and digital communications [15, 16]. We use two signal processing approaches to complete the matching: cross-correlation and maximum absolute error. For our application, cross-correlation is extremely simple.

Assume that the columns of the matrix $\mathbf{C}$ (the signature database) contain the frequency response vectors (taken across a wide band of frequencies around the expected resonant frequency) at each of the m temperatures of interest, i.e., $\mathbf{C} = [\mathbf{C}_1, \mathbf{C}_2, \dots, \mathbf{C}_m]$. Suppose that the measured frequency response at an unknown temperature is contained in the column vector $\mathbf{r}$. We create the $1 \times m$ decision statistic

$$\mathbf{d} = \mathbf{c}^T \mathbf{r}, \quad (2)$$

Our temperature estimate (index) is simply given as the index of the maximum component in $\mathbf{d}$, call it $\hat{\mathbf{m}}_{CC}$ for the cross-correlation algorithm. We look up the temperature that corresponds to this index and we have our temperature estimate. Importantly, our method does not rely on any particular physical material model. It only requires that the wideband frequency response change in some way across frequencies. When material properties change due, for example, to heating and cooling cycles, we need only update our database matrix $\mathbf{C}$ with new signatures at known temperatures. Thus, our approach is *robust* and *adaptive*.

Our minimum absolute error method simply finds the signature which has the minimum absolute error difference relative to $\mathbf{r}$. Let $\mathbf{s}_i = |\mathbf{r} - \mathbf{C}_i|$, $i = 1, 2, \dots, m$ be the absolute difference vector between $\mathbf{r}$ and the i^{th} signature. Let $\bar{s}_i$ be the sum of the elements of $\mathbf{s}_i$. The minimum absolute error temperature (index) estimate can be simply written as $\hat{\mathbf{m}}_{AE} = \arg \min_i \bar{s}_i$.

Both temperature index estimates $\hat{\mathbf{m}}_{CC}$ and $\hat{\mathbf{m}}_{AE}$ can be computed easily using Matlab or any computing language. To improve performance, we used a sliding window optimization technique to determine the best band of frequencies to use (within the wideband response) for temperature estimation. We can determine this *a priori* by experimentation before the algorithm is used for temperature estimation. Instead of analyzing the entire spectrum of data, we use overlapping windows of various sizes and center frequencies and choose the window width and center frequency that maximizes performance. The window size and the position of the window are both variable and can be optimized based on its performance. This example shows a window size of 5

and changing the window position from 10.1 MHz in the first case to 10.2 MHz in the second case and so on. After running the methods through all these cases, the new algorithm will output all cases which give the correctly predicted temperature. In this work, the algorithm with window sizes of 5, 10, 50, 100, 500, 1000, 2000 and 2400 were used to predict the temperature from the wireless signal response irrespective of its resonant frequency.

2.3. Fabrication of the all-ceramic wireless LC resonator sensors

As previous stated, the active sensor circuit was composed of a conductive ceramic composite deposited as a 2D LC circuit onto Al_2O_3 substrates. The conductive ceramic composite was based upon the lanthanum nickelate (La_2NiO_4) composition, which was specifically synthesized in this work using a solid-state process. Reagent grade lanthanum carbonate hydrate ($\text{La}_2(\text{CO}_3)_3 \cdot x\text{H}_2\text{O}$) and nickel oxide (NiO) (Alfa Aesar, Tewksbury, MA) were used as the precursors. The lanthanum carbonate and nickel oxide were weighed according to the nominal molar composition of La_2NiO_4 . For simplicity, La_2NiO_4 will be inferred as LNO in the rest of the paper. The powders were dry milled in a roll mill for 2 h and mixed thoroughly in an attrition mill for 2 h with ethanol as the dispersant. The mixture was dried thoroughly before solid-state calcination at 1400°C for 4 h in a muffle furnace (MTI Corp, Richmond, CA). X-ray diffraction (XRD, X'Pert Pro Panalytical, Westborough, MA) with Al K_α was used to identify the phase composition of the synthesized LNO in order to verify the phase purity of the calcined LNO.

Initially, pure LNO was deposited on an Al_2O_3 substrate (MTI Corp, Richmond, CA) by a screen-printing technique to test the wettability and adhesion of LNO at high temperature. After pyrolysis at 1200°C for 2 h, the LNO thick film delaminated from the Al_2O_3 substrate. To mitigate the adhesion issue, composites of different volume fractions of LNO- Al_2O_3 composites were synthesized and studied. The LNO- Al_2O_3 composite powders with two different volume fractions (50 and 60 vol%) were synthesized by roll milling LNO and fine Al_2O_3 (99.8%, $8.6 \text{ m}^2/\text{g}$ SSA; Almatix, Leetsdale, PA).

The composites were pressed into 25 mm diameter pellets by a uniaxial press and sintered at 1200°C for 2 h. Further long-term isothermal phase stability analysis was performed. The sintered composites were characterized by XRD and electrical conductivity before fabricating the wireless sensor. The results were discussed in the following section. The thick film electrical conductivity of the 50-50 and 60-40 LNO-Al₂O₃ composites were tested by four-point conductivity method. For XRD analysis, the samples were prepared from a bulk pellet of the composition. The conductivity measurements were completed in a tube furnace (Sentro Tech, Strongsville, OH) and *in situ* electrical acquisition were recorded by a digital multimeter (DMM, Keithley Instruments, Cleveland, OH).

The wireless LC resonators were fabricated by using a combination of photolithography (fig. 3a) and micro-casting process (fig. 3b). The flowchart of two-step fabrication process is shown in fig. 3 (a & b). In this case, a conventional photolithography process was used to create micro-molds for the IDC and an inductor circuit pattern on the Al₂O₃ substrate (fig. 3a). A 35 µm of NANO^{TM} SU-8 25 (Microchem, Westborough, MA) photoresist was spin-coated onto the Al₂O₃ substrate and baked on a hot plate as per the manufacturer's standards. A high-resolution quartz photomask (Photo Sciences, Torrance, CA) was used to transfer the LC resonator pattern to the Al₂O₃ substrate, as discussed in the sensor design section. The substrates were developed with SU-8 developer (Microchem, Westborough, MA) for 40 – 60 s and washed several times with isopropyl alcohol (VWR, Radnor, PA).

The second step in the fabrication process is shown in fig. 3b. The functional ceramic ink was synthesized by dispersing 40 vol% of 60-40 LNO-Al₂O₃ composite powder in an organic ink vehicle (63-2 vehicle, Johnson-Matthey, USA). A commercially available squeegee blade with a shore hardness of 70 durometers (Screen Printing Supply Store, NY) was used to cast the ink into the cavities of the micro-molds. The micro-casted substrates were dried on a hot plate at 90°C for 3-5 min. and then sintered at 1200°C for 2 h. No additional lift-off process was performed. The photoresist layer was burned away *in situ* during the thermal processing step through pyrolysis in air. The patterned sensor

substrates were cleaned by sonicating in an ultrasonic bath for ~1 min. Pt interconnects were made using Pt ink and stabilized by sintering at 1050°C for 24 h. The microstructure of the sensor before and after different thermal cycles was analyzed using scanning electron microscopy (SEM, JOEL JSM-7100F).

(a) Photolithography Process to Create Micro-molds

(b) Micro-casting Process to Fabricate Wireless Sensor

Fig. 3. Flowchart showing the step-by-step fabrication process of the passive wireless sensor: (a) photolithography process to create the micro-molds, (b) micro-casting of the LNO – Al_2O_3 ink within the micro-molds using a squeegee blade (shore hardness – 70 durometer).

2.4. Wireless characterization setup

The pure passive functionality of the sensors was not evaluated in this work but were reserved for the follow-up publications. In this paper, the sensor concept was tested by hardwiring the sensor with Pt interconnects as explained in the previous section. A pair of identical resonators (as shown in fig. 4a) were used to characterize the wireless signal response where one resonator was connected to the signal generator (actual working sensor) and the other to the spectrum analyzer (interrogator antenna). Identical resonators were used to maximize the mutual inductance between the sensor and the interrogator, which leads to better coupling and increased signal efficiency. The interrogator can also be replaced with a commercial PCB antenna with a frequency range between 10 - 100 MHz. However, the RF signal from the interrogator needs to be amplified to have a higher signal-to-noise ratio. The fabricated wireless sensors were characterized from 500 – 1000°C in a muffle furnace (MTI Corp, Richmond, CA) at ambient atmosphere with *in situ* electrical data acquisition using a HP E4433B signal generator (now Keysight, Santa Rosa, CA) and a RSA306B USB spectrum analyzer (Tektronix, Beaverton, OR). The sensors were placed ~10 cm apart and supported with an alumina testbed with the inductors facing each other in order to maximize the coupling efficiency. The sensor connected to the signal generator acts as the interrogator antenna which will sweep the frequency from 10 to 80 MHz with 175 kHz·s⁻¹ sweep rate. The RF signal would excite the sensor connected to the spectrum analyzer (signal receiving sensor) through inductive coupling between the interrogator antennas. This induced inductance causes a current to flow through the sensor connected to the spectrum analyzer. The receiving sensor re-radiates the RF signal. The re-radiated RF signal is recorded by the signal analyzer. An example of this frequency response by the receive sensor can be seen on the monitor on the right of fig. 4b.

The initial characterization of the sensor was performed by measuring the frequency response from 500 to 1000°C with an increment of 100°C. The frequency response is logged multiple times

for a given temperature and the average of the data points were used in further analysis. The data from this initial run will be referred to as a signature database since the temperature readings were known by measuring with an external thermocouple. In order to characterize the sensor, the temperature was cycled between 500 and 1000°C with *in situ* wireless signal acquisition as explained above. The sensor response from the unknown temperature is compared to that of the signature database to estimate the unknown temperature by the signal processing algorithms.

Fig. 4. (a) Passive wireless sensor (before wiring) used for wireless characterization; (b) Passive wireless characterization setup with sensors (inside the furnace) connected to a signal generator and analyzer.

3. Results and discussion

3.1. Phase development in the electroceramic composite

The LNO and its composites with Al_2O_3 were used as the conductive material to fabricate the wireless resonators. Any change in the microstructure and/or chemical composition during the

sintering process (or continuous thermal cycle) may alter the electrical property of the conductive composite. This change in electrical property may reflect in the wireless response as additional noise or frequency shift. Therefore, it is important to understand the chemical/phase stability of the LNO- Al_2O_3 electroceramic composites at high temperature printed onto the given substrate. The phase analysis study was performed by XRD on the pure LNO and LNO- Al_2O_3 composites to understand the phase development and secondary phase formation in the ceramic composite. The XRD phase analysis of the LNO particles calcined at 1400°C was performed to confirm there are no secondary phase formation during the calcination process. Fig. 5 shows the XRD pattern of single phase LNO which has a tetragonal crystal structure with a space group of $I4/mmm$ [17]. The single phase tetragonal LNO with a pure K_2NiF_4 structure was reported to be a good electronic semi-conductor for high temperature electroceramic applications [17-20].

Fig. 5. XRD pattern of LNO synthesized by solid state reaction of lanthanum carbonate and nickel oxide sintered at 1400°C for 2 h in air.

Initially, the pure single phase LNO was deposited on the Al_2O_3 substrate using screen printing technique and sintered at 1200°C . The post sintered monolith showed delamination of the LNO from the Al_2O_3 substrate. Among the several factors affecting the adhesion of the LNO on the Al_2O_3 substrate at high temperature, coefficient of thermal expansion (CTE) and coarsening of

LNO are the significant effects. The CTE of Al_2O_3 is $8.1 \times 10^{-6} \text{ K}^{-1}$; however, the LNO composition has a CTE of $\sim 13.6 \times 10^{-6} \text{ K}^{-1}$, where the difference in the CTE is $\sim 5.5 \times 10^{-6} \text{ K}^{-1}$ [18]. This difference in the CTE is one of the factors leading to delamination of the LNO layer from the Al_2O_3 substrate.

Fig. 6. XRD pattern of (a) 50-50 LNO- Al_2O_3 composite showing the presence of LaAlO_3 and NiAl_2O_4 phase, (b) 60-40 LNO- Al_2O_3 composite showing the presence of LaAlO_3 , NiAl_2O_4 , and NiO phase undergone various isothermal loading cycles.

To mitigate the delamination of the LNO layer on the Al_2O_3 substrate, the composites of LNO- Al_2O_3 with varying percentages of LNO was synthesized and characterized by XRD. The XRD patterns of 50-50 and 60-40 LNO- Al_2O_3 composites that were sintered at 1200°C for 2 h and annealed at 1050°C for 24 h, 48 h, 72 h, and 96 h are shown in fig. 6(a & b), respectively. After sintering the composite, consisting of 50 vol% of LNO in Al_2O_3 , the XRD pattern showed that the LNO reacts with Al_2O_3 with in the intimate composite. The reaction forms two new phases, namely a perovskite lanthanum aluminate (LaAlO_3) and a spinel nickel aluminate (NiAl_2O_4). However, the 60 vol% LNO in Al_2O_3 composite forms nickel oxide (NiO) phase in addition to the LaAlO_3 and NiAl_2O_4 phase. The phase transformation/degradation of the La_2NiO_4 phase was reported by Schrödl et al. in the presence of chromium (Cr) [19, 20]. Schrödl et. al. study showed

that the La_2NiO_4 phase was stable up to 950°C and was not greatly poisoned by chromium around this temperature regime [19]. Schrödl et. al. showed evidences that the transport of Cr cations along the grain boundaries plays significant role in the degradation of La_2NiO_4 phase [19]. A similar reaction mechanism might be undergone in the case of LNO- Al_2O_3 composites, where the Al^{3+} cation diffuse through the grain boundary and react with the La_2NiO_4 phase. In the 60-40 composite, the formation of the three phases due to excess precipitation of nickel from the LNO phase and only a certain percentage of Al_2O_3 is available for the nickel to react to form NiAl_2O_4 phase and the rest reacts with oxygen (from the atmosphere) to form NiO phase. However, in the case of 50-50 composite, the molar ratio of the elements is exact to form only two phases, LaAlO_3 and NiAl_2O_4 . Further high temperature annealing was completed on these composites in air for different isothermal hold times in order to better understand rate of continued reaction at these temperatures, which again would be important to understand for extended sensor testing. As shown in fig. 6(a & b), the XRD pattern over this time scale shows that after the initial thermal processing step, there is no further phase transformation/degradation.

Fig. 7. Rietveld refinement results of (a) 50-50 and (b) 60-40 LNO- Al_2O_3 composites annealed at 1050°C for 24h (solid red lines = fitting curves; black dots = experimental XRD data).

Rietveld refinement was performed on the 50-50 and 60-40 LNO-Al₂O₃ composites that were sintered at 1200°C for 2 h and annealed at 1050°C for 24 h, 48 h, 72 h, and 96 h in order to calculate the weight fraction of each phase present in the composite and to understand the phase stability over a long period of isothermal loading at operating temperature (1050°C). Fig. 7(a & b) shows the Rietveld refinement results of the 50-50 and 60-40 composites annealed at 1050°C for 24 h, respectively, where the solid lines represent the fitting curve and the black dots denotes the experimental data from XRD. The quality of the Rietveld refinement can be defined by the goodness of the fit (sig) and weighted profile R-factor (R_{wp}). The sig and R_{wp} of the analysis are ranging from 1.2 – 2.1 and 4.0 – 5.2, respectively [21]. The volume percentages of the phases present in the 50-50 and 60-40 composite for varying annealing time is shown in table 2 and 3. It can be inferred from table 2 and 3 that after the initial sintering there is neither a significant change in the amount of the phases nor further phase transformation.

Table 2. Rietveld analysis of 50-50 LNO-Al₂O₃

Phase	Wt%				
	1200°C – 2h	1050°C – 24h	1050°C – 48h	1050°C – 72h	1050°C – 96h
LaAlO ₃	51.64	51.15	49.28	49.90	47.32
NiAl ₂ O ₄	48.36	48.85	50.72	50.10	52.68

Table 3. Rietveld analysis of 60-40 LNO-Al₂O₃

Phase	Wt%				
	1200°C – 2h	1050°C – 24h	1050°C – 48h	1050°C – 72h	1050°C – 96h
LaAlO ₃	36.30	35.78	34.51	33.25	33.71
NiAl ₂ O ₄	30.86	33.41	30.05	31.09	31.26
NiO	32.84	30.81	35.43	35.67	35.03

3.2. Electrical characterization of the ceramic composites

The electrical property of the LC resonator such as capacitance and inductance depend on the electrical conductivity of the electroceramic composite, and these conductive lines forming the LC sensor must remain consistently conductive during operation. Therefore, the conductive properties of these materials must remain stable over the duration of high-temperature operation; otherwise, the intrinsic electrical signature may alter which would lead to a misinterpretation of a temperature or mechanical event. So, the electrical conductivity of the 50-50 and 60-40 LNO- Al_2O_3 composites was tested from 500 to 1200°C. Fig. 8(a & b) shows the electrical conductivity of the 50-50 and 60-40 LNO- Al_2O_3 composite between this temperature range. The conductivity increases as a function of temperature for both the composites which indicates the semiconducting nature of the composites. It can be seen from fig. 8(a) that the conductivity of 50-50 LNO- Al_2O_3 composite has two different slopes and there is a transition at temperature $\sim 600^\circ\text{C}$. Further analysis was performed by taking the first order derivative of the conductivity with respect to temperature. The analysis shows that the conductivity has two linear regimes that is from 500 to 620°C, and 620 to 1100°C. Recalling from the XRD analysis discussed in section 3.1, the 50-50 LNO- Al_2O_3 composite has two different phases namely, LaAlO_3 and NiAl_2O_4 . Previous reports on LaAlO_3 stating that it is a high- κ (but weak dielectric) material [22]. Suzuki et al. [23] report showing the band gap of LaAlO_3 is 6.5 eV which clearly proves it is an electronic insulator. Materials with such a large band gap will stay as a stable insulator even at high temperature [23]. Kou et al. [24] study shows NiAl_2O_4 spinel phase is a p-type semiconductor under similar operating conditions. The report also shows that the conductivity mechanism of the NiAl_2O_4 spinel phase which is due to the formation of nickel vacancies as a function of temperature. The temperature dependent conductivity response is also similar to the one presented in this work. Based on the evidences reported by Kou et al. [24], it can be confirmed that the electrical

conductivity of the 50-50 LNO- Al_2O_3 composite is due to the presence of NiAl_2O_4 spinel phase. However, the conductivity of the composite is lower than the conductivity of the pure NiAl_2O_4 phase since the composite is a combination of an insulating and a semi-conducting phase [24].

Fig. 8. Thermoelectric conductivity of (a) 50-50 LNO- Al_2O_3 and (b) 60-40 LNO- Al_2O_3 composites printed on an Al_2O_3 substrate as a function of temperature from 500 to 1200°C.

Fig. 8(b) shows the electrical conductivity of the 60-40 LNO- Al_2O_3 composite. Similar analysis was performed on the electrical conductivity data of the 60-40 LNO- Al_2O_3 composite. The conductivity of the 60-40 LNO- Al_2O_3 composite also shows two linear regions between 500 – 800°C and 800 – 1200°C. Recalling from the Rietveld analysis in section 3.1, the 60-40 LNO- Al_2O_3 composite has ~34 wt% of NiO and 31 wt% of NiAl_2O_4 . Since the wt% of the semiconducting phases in the 60-40 LNO- Al_2O_3 is ~65%, the conductivity of the 60-40 LNO- Al_2O_3 composite is an order magnitude higher than the 50-50 LNO- Al_2O_3 composite. The electrical conductivity of the NiAl_2O_4 was discussed earlier in this section. Mitoff et al. [25] reported on temperature dependent electrical conductivity of NiO, where NiO is also a p-type semiconductor and the mechanism of electrical conductivity is due to the formation of nickel vacancies as a function of temperature. Feinleib et al. [26] performed first principle calculations on the band structure of pure NiO to showcase temperature electrical conductivity of the NiO. The

results also corroborate the experimental values reported by Mitoff et al. [25]. The temperature dependent electrical conductivity behavior presented in fig. 8(b) is similar to the work reported by Mitoff et al. [25]. It is desirable to have higher conductive electroceramic composite for the sensor fabrication in order to improve the signal response as well as reduce signal-to-noise ratio. Owing to the microstructural stability and increased electrical conductivity, the 60-40 LNO- Al_2O_3 composite was chosen to fabricate and characterize the wireless sensor.

3.3. Microstructure of the ceramic LC resonator

If there is a microstructural change such as densification or grain growth as a function of thermal cycles, then the electrical conductivity (and wireless response) of the sensor composite will be altered. The microstructure of the LC resonator was analyzed by using SEM in order to understand the effect of grain growth or coarsening during the thermal operation cycles up to 1200°C. Microstructural stability is very important for repeatable and long-term sensor operation.

Fig. 9. SEM micrograph of 60-40 LNO- Al_2O_3 composite LC sensor circuit (a, c) sintered at 1200°C for 2 h, (b, d) thermal cycles at 1050°C for cumulatively 240 h (24 h + 48 h + 72 h + 96 h). Fig.s (a) and (b) shows the low magnification images whereas, (c) and (d) shows the high magnification comparison of 60-40 LNO- Al_2O_3 composite LC sensor circuit.

Similar sensors (LC circuits) were fabricated and cut into 10×10 mm squares to perform SEM analysis which underwent the same thermal cycle as per the wireless sensor. One of the samples were imaged after sintering for 2 h at 1200°C and a similar sample was imaged after undergoing several thermal cycles during the wireless characterization. Fig. 9 (a, c) shows the SEM microstructure of the LC circuit which underwent only the sintering cycle at 1200°C for 2 h. The micrograph reveals the necking and percolation of the grains was well pronounced. Fig. 9 (b, d) shows the SEM micrograph of the LC circuit which underwent several thermal cycles during the wireless characterization. The micrograph is similar to the sensor before the wireless characterization. The micrograph was analyzed by computation image analysis, and the average grain size ranges from $0.5 - 3 \mu\text{m}$, which corroborates that there is relatively low coarsening or other microstructural changes during the operation cycle. Also, from image analysis, the porosity within the printed LC circuit ranges from 50 - 60%, which also indicates that there is relatively no densification occurring during the repeated thermal cycles. It is evident from the microstructural analysis that the microstructure is stable after several cycles of thermal loading.

Fig. 10 (a-d) represents the optical microscope photographs and SEM micrographs (e-h) of the 60-40 composite micro-casted on the Al_2O_3 substrate. The LC sensor pattern with different magnification was shown in fig. 10 (a-d) which shows that there is no short circuit or macroscopic defects such as a discontinuity in the IDC and inductor pattern. It also shows that there is no residue from the micro-casting process after sintering and cleaning of the patterned substrate in an ultra-sonication bath. Fig. 10 (e-h) shows the SEM micrograph of one of the IDC/inductor

patterns. Fig. 10 (h) shows the magnified image of the sensor material which has a porous structure as discussed above. Fig. 10 (g) shows the magnified image of the dense Al_2O_3 substrate. A thorough optical inspection was completed in order to make sure there are no defects in the IDC/inductor. If there is a defect such as delamination of the electroceramic composite, it would lead to an open circuit where the entire sensor becomes inoperable. If there is a short-circuit between two lines by means of a residue or improper cleaning during the pre- or post-processing of the micro-casting process, it will lead to electrical fluctuation and reduced capacitance or inductance.

Fig. 10. (a-d) Optical microscope image of the LC sensor circuit fabricate with 60-40 LNO- AlO_3 composite at different magnification showing that there are no defects after sintering at high

temperature; (e-h) represents the SEM micrograph distinguishing IDC/inductor pattern from the Al_2O_3 substrate.

3.4. Wireless characterization

In literature [8-15] several aspects of the wireless signal processing techniques were utilized to analyze the output signal from the RF sensors. A general trend in the signal processing of RF based sensor relies on the change in impedance (S_{11} parameter) parameter or shift in the phase angle ($^\circ$) with respect to the sensing medium [8-15]. The reported literatures were focused on developing RF based wireless sensors to operate at a particular frequency known as resonant frequency which shifts as a function of temperature (or any other measuring parameter). The resonant frequency was predetermined by computationally modelling the effective LC circuit. The experimental results were compared with the model to showcase the performance and accuracy of the sensor. Tan et al. [14] and several other researchers reported passive wireless sensor developed by low temperature co-fired ceramic (LTCC) technique based on the aforementioned signal processing technique. Instead of analyzing the conventional S_{11} parameter or the phase angle of the antenna, we looked at the received signal strength (RSS) from the sensor as a function of temperature. The RSS works similar to the other types of sensing mechanisms, but it spans over a wide range of frequency (10 – 80 MHz) instead of focusing at a narrow bandwidth. To understand the operating frequency bandwidth of the sensors, we followed a similar approach to model an effective LC circuit using the Ansys Maxwell software which showed the maximum intensity of the RSS was centered at 13.56 MHz assuming that the electrodes were metallic. Fig. 11 shows the wireless response of the sensor from 500 – 1000°C centered at 13.56 MHz. The peak shift in RSS with respect to the temperature is not well pronounced. For example, the RSS for temperatures 900°C and 1000°C overlaps at 13.56 MHz rendering the wireless signal useless to distinguish the temperature change.

Fig. 11. Magnified frequency response showing the sensor response centered at 13.56 MHz.

Generally, the sensors presented in literature were tested under an ideal operating environment, where the wireless signal from the sensor was captured in an electrically insulated furnace/system. This environment is not representative of potential application of these sensors. In addition, in the case of the temperature regimes targeted in this work, this ideal environment would be difficult to create inexpensively, where it would be expected that there would be electrical/magnetic interferences from various electrical components in the high temperature system. In order to check the contribution of the background signal interference with the wireless response of the sensor, we collected the background signal from the furnace without a sensor. Fig. 12 shows the average of the background signal from 500 – 1000°C. The RSS of the background noise has a signal strength of -45 dBm from 10 – 25 MHz and -60 dBm from 25 – 80 MHz. From this spectrum, it appeared that the background noise showed negligible effect on the sensor response at 13.56 MHz, since the RSS at 13.56 MHz lies above -15 dBm.

Fig. 12. Wide-Band frequency response of the interrogator antenna showing the background noise at high temperatures fabricated with 60-40 LNO- Al_2O_3 material composition.

Other possible contributions which causes the attenuation and multiple peak shifts across the wide bandwidth of the spectrum can be related to the electrical properties of the interconnects and the LC electrodes. Although no available reports or model are showing the effect of electrical conductivity of the electrodes (capacitor and inductor electrodes) on the wireless response of an RF-based passive wireless sensor, it is well known that the higher conductivity results in the better inductive coupling between the sensor and the interrogator antenna. The previous RF-sensors reported in the literatures at elevated temperatures ($\sim 100\text{-}500^\circ\text{C}$) were composed of metallic electrodes like copper, silver, platinum, and metal/ceramic composites. Due to this reason, the reported wireless sensors (LC resonator) in the literature behaves similar to their computational model [9-17]. But the electrical conductivity of the semi-conducting electroceramic composite used in this work is significantly lower than the metals. This may have caused an attenuation in the RSS and shifted the peak to a different location in the spectrum. Several parameters such as parasitic electrical noise from the testing equipment, electrical interconnect at high temperature, and maintaining both the sensor and interrogator antenna at the hot zone may have led to the multiple peaks in the spectrum.

Since there are noticeable shifts recognized at different locations of the spectrum, the entire bandwidth of the spectrum (10 - 80 MHz) was considered for further analysis. Wireless signal processing was performed in two steps for the entire bandwidth of the spectrum: 1) a temperature signature was created by averaging the frequency response from the wireless sensor over ten cycles while simultaneously measuring the temperature with an external thermocouple, 2) a similar approach was completed to collect the signal response from the sensor at each characterization temperature but without using an external thermocouple and labeled as unknown temperature readings. There are three measurements taken for each sensor, where the first measurement was collected with the direct relationship to the temperature with an external thermocouple. The sensor was cycled two more times and the response was recorded. In this work three different sensor/antenna pairs (hereafter, named as *Sensor 1*, 2, and 3) were tested to complete the wireless characterization, where the sensors pairs were fabricated using in the same fashion with the same materials.

Fig. 13 (a & b) shows the broadband wireless response of the first sensor/antenna pair (*Sensor 1*) during the second and third thermal cycles, respectively. The first cycle was completed with an external thermocouple to measure the response with respect to temperature. In the second and third cycles shown in this Fig. 13, an external thermocouple was not used, and these measurements were termed as “unknown” measurements (labelled as *Unk 1* and *Unk 2* measurement). The first thermal cycle is represented in the fig. 13 and labelled as *Sig* (i.e the temperature signature). Both the *Unk 1* and *Unk 2* macroscopically looked the same with peak shifts observed at various location of the spectrum. For *Sensor 1*, we used Pt wire to connect the sensor shown in fig. 13 to the signal generator. The Pt wire was bonded on to the contact pads of the sensor with an ink synthesized by Pt particles with ~1-3 μm in diameter. The pure metallic Pt ink connection began to delaminate after undergoing two thermal cycles from 500 to 1000°C. The

delamination arises from the coarsening of the Pt particles at high temperature. This may also be a contributing factor to the change in the shape of the RSS. As inferred from fig. 13 (a & b), there is a change in peak shape around 25 MHz. This may have been caused by the slight delamination of Pt interconnect at the contact pads because all the other parameters were kept the same during the wireless characterization.

Fig. 13. Wide-Band frequency response of Sensor 1 fabricated with 60-40 LNO-Al₂O₃ material composition from 500 – 1000°C.

As discussed previously, two additional sensor pairs (*Sensor 2* and *Sensor 3*) were fabricated and characterized in the same fashion as that of the first sensor pair showed in fig. 13. In order to see the effect of sensor electrical contact to the Pt wire on the wireless response of the sensor, we

replaced the Pt ink with the same composition as the sensor design (with LNO-Al₂O₃ ink). The LNO-Al₂O₃ showed better adhesion to the contact pads as the contacts were made with the same material system. The wireless response from the second and third sensor pairs (*Sensor 2* and *Sensor 3*) are shown in fig. 14 (a, b) & (c, d), respectively. It can be inferred from fig. 13 and 14 that there is a difference in the wireless response of the sensor tested with Pt and LNO-Al₂O₃ interconnects at the contact pads, respectively. It is evident from above analysis that the sensor connection plays an important role in that the wireless response of the wireless response (in this case, where both sensor and integrator antennae are physically wired and in the hot-zone). Additionally, the wireless response of the sensors shown in fig. 14 (a, b) & (c, d) is similar with a minimal change in the shape of the RSS, which may be due to the previous discussed materials, electrical and/or external parameters. For instance, small changes in microstructure and chemistry may have occurred during processing, and/or changes in the potential interconnection, which could have resulted in the intensity changes seen in the response. To identify these effects, it may take materials scientists extensive time to better understand and provide a solution. Therefore, a signal processing method should be applied that permits an adaptive analysis of the sensor signal, regardless of initial or *in situ* changes in the sensor response.

To further analyze the large dataset, we used a sliding window optimization technique to determine the best band of frequencies to use (within the wideband response) for temperature estimation using the Matlab software as discussed in section 2.2. The sliding window technique utilizes a modular and piecewise approach to compare a set of frequencies instead of the entire spectrum. The sliding window technique used in this work had a window size ranging from 5 to 2400. The window size determines the set of frequencies that are taken into account for the analysis. For instance, a window size 5 represents the first five data points considered for analysis and the algorithms find the correlation for that particular data set before moving to the consecutive

(next 5) data points. A similar approach was performed for the other window sizes to determine the best matching window size for each data sensor pair. The optimal window size was chosen to yield the best match between the signature database and the unknown.

Fig. 14. Wide-Band frequency response of Sensor 2 (a & b) and Sensor 3 (c & d) pairs fabricated with 60-40 LNO- Al_2O_3 material composition from 500 – 1000°C.

After defining the window size, the two signal processing algorithms were used to match a *Sig* with *Unk 1* and *Unk 2* for each sensor pair. In particular, the cross-correlation algorithm looks for the signature (*Sig*) that has maximum similarity with the unknowns (*Unk 1*, *Unk 2*). On the other hand, the minimum absolute error algorithm looks for the minimum absolute difference between the temperature signature (*Sig*) and the unknowns. In order to compute the cross-correlations and

absolute differences easily, a database of signature waveforms (Sig) at each temperature of interest are initially created offline, before real-time data collection. These waveforms are placed as column vectors in the matrix $\mathbf{C}$ as discussed in Section 2.2. The first column of $\mathbf{C}$ represents the signature for 500°C. The second column represents the signature for 600°C, and so on to 1000°C. We denote each column separately using a subscript, i.e.,

$$\mathbf{c}_{500^\circ\text{C}} = \begin{bmatrix} c_{500^\circ\text{C}}^{10.0} \\ c_{500^\circ\text{C}}^{10.1} \\ c_{500^\circ\text{C}}^{10.2} \\ \vdots \\ c_{500^\circ\text{C}}^{80.0} \end{bmatrix}_{700 \times 1} \quad \dots \dots \dots \mathbf{c}_{1000^\circ\text{C}} = \begin{bmatrix} c_{1000^\circ\text{C}}^{10.0} \\ c_{1000^\circ\text{C}}^{10.1} \\ c_{1000^\circ\text{C}}^{10.2} \\ \vdots \\ c_{1000^\circ\text{C}}^{80.0} \end{bmatrix}_{700 \times 1} \quad (3)$$

where, $c_{500^\circ\text{C}}^{10.0}$, $c_{500^\circ\text{C}}^{10.1}$, $c_{500^\circ\text{C}}^{10.2}$, ..., $c_{500^\circ\text{C}}^{80.0}$ represent the RSS at frequencies 10.0, 10.1, 10.2, ..., 80.0 MHz at 500°C. Thus, one matrix with 6 columns contains the signatures (Sig) from 500°C to 1000°C. The combined column vectors for all the temperature signatures from 500 – 1000°C is written as $\mathbf{C}$,

$$\mathbf{C} = [\mathbf{c}_{500^\circ\text{C}} \quad \mathbf{c}_{600^\circ\text{C}} \quad \mathbf{c}_{700^\circ\text{C}} \quad \mathbf{c}_{800^\circ\text{C}} \quad \mathbf{c}_{900^\circ\text{C}} \quad \mathbf{c}_{1000^\circ\text{C}}]_{700 \times 6} \quad (4)$$

Similarly, there are 6 column vectors in a matrix for each dataset represented as *Unk 1* and *Unk 2* which is represented as $\mathbf{r}$ and can be written as,

$$\mathbf{r}_{500^\circ\text{C}} = \begin{bmatrix} r_{500^\circ\text{C}}^{10.0} \\ r_{500^\circ\text{C}}^{10.1} \\ r_{500^\circ\text{C}}^{10.2} \\ \vdots \\ r_{500^\circ\text{C}}^{80.0} \end{bmatrix}_{700 \times 1} \quad \dots \dots \dots \mathbf{r}_{1000^\circ\text{C}} = \begin{bmatrix} r_{1000^\circ\text{C}}^{10.0} \\ r_{1000^\circ\text{C}}^{10.1} \\ r_{1000^\circ\text{C}}^{10.2} \\ \vdots \\ r_{1000^\circ\text{C}}^{80.0} \end{bmatrix}_{700 \times 1} \quad (5)$$

The column vectors for the unknowns from 500 to 1000°C can be written as,

$$\mathbf{R} = [\mathbf{r}_{500^\circ\text{C}} \quad \mathbf{r}_{600^\circ\text{C}} \quad \mathbf{r}_{700^\circ\text{C}} \quad \mathbf{r}_{800^\circ\text{C}} \quad \mathbf{r}_{900^\circ\text{C}} \quad \mathbf{r}_{1000^\circ\text{C}}]_{700 \times 6} \quad (6)$$

The matrix $\mathbf{R}$ is multiplied with the transpose matrix of the temperature signatures ($\mathbf{C}^T$) in order to derive the decision statistics ($\mathbf{D}$) which is represented as,

$$\mathbf{D} = \mathbf{C}^T \times \mathbf{R} \quad (7)$$

$$\mathbf{C}^T \times \mathbf{R} = \begin{bmatrix} c_{500^\circ C} \\ c_{600^\circ C} \\ c_{700^\circ C} \\ c_{800^\circ C} \\ c_{900^\circ C} \\ c_{1000^\circ C} \end{bmatrix}_{6 \times 700} \times [r_{500^\circ C} \quad r_{600^\circ C} \quad r_{700^\circ C} \quad r_{800^\circ C} \quad r_{900^\circ C} \quad r_{1000^\circ C}]_{700 \times 6} \quad (8)$$

$$\mathbf{D} = \begin{bmatrix} c_{500^\circ C}^T \times r_{500^\circ C} & \cdots & c_{500^\circ C}^T \times r_{1000^\circ C} \\ \vdots & \ddots & \vdots \\ c_{1000^\circ C}^T \times r_{500^\circ C} & \cdots & c_{1000^\circ C}^T \times r_{1000^\circ C} \end{bmatrix}_{6 \times 6} = \begin{bmatrix} d_{500^\circ C-500^\circ C} & \cdots & d_{500^\circ C-1000^\circ C} \\ \vdots & \ddots & \vdots \\ d_{1000^\circ C-500^\circ C} & \cdots & d_{1000^\circ C-1000^\circ C} \end{bmatrix}_{6 \times 6} \quad (9)$$

The elements of the decision statistics matrix ($\mathbf{D}$) is represented as $d_{500^\circ C-500^\circ C}$, $d_{500^\circ C-600^\circ C}$, ..., $d_{1000^\circ C-900^\circ C}$, $d_{1000^\circ C-1000^\circ C}$ which is obtained by processing the elements in $\mathbf{C}^T$ and $\mathbf{R}$ with the cross-correlation and minimum absolute error algorithms. The elements in the matrix $\mathbf{D}$ represents a numerical value which is considered as a “score” to evaluate the goodness of the match. The first column of the matrix $\mathbf{D}$ represents the data obtained by evaluating the *Sig* measured at $500^\circ C$ with all the unknown temperatures (500 to $1000^\circ C$). A similar evaluation was performed on the rest of the temperatures (600 – $1000^\circ C$) in the columns two to six. As inferred from the Eq. (9), the diagonal elements in the matrix $\mathbf{D}$ represents data evaluated for the temperature signatures and the unknowns at same temperature, i.e., $d_{500^\circ C-500^\circ C}$ represents the evaluation of the wireless response of *Sig* and *Unk 1* at $500^\circ C$, $d_{600^\circ C-600^\circ C}$ represents the data for *Sig* and *Unk 1* at $600^\circ C$ and so on. For a good match, the diagonal elements in the matrix $\mathbf{D}$ must be the larger than the non-diagonal elements. If any of the non-diagonal elements is greater than the diagonal elements, then the sensor response at that particular frequency range (or window size) is rendered incorrect. Additionally, this method of processing wireless signals does not require a user defined threshold because the score provided to each element in matrix $\mathbf{D}$ is relative and eliminates a hard threshold

limit. The non-diagonal elements simply represent how distinguishable the unknown waveforms with respect to the temperature signatures. The higher difference between the diagonal and the non-diagonal elements represents the temperature signatures and the unknowns are more distinguishable from the other temperatures.

Table 4. Total number of possible temperature signature matches for all the three sensor pairs with respect to the window size.

Sample/Unknown	Number of matches for each window size							
	5	10	50	100	500	1000	2000	2400
Sensor 1-Unk 1	9	8	10	12	10	6	4	3
Sensor 1-Unk 2	12	12	8	8	9	7	4	4
Sensor 2-Unk 1	9	10	10	9	9	10	10	10
Sensor 2-Unk 2	11	11	11	12	12	11	10	10
Sensor 3-Unk 1	11	11	10	10	12	11	10	10
Sensor 3-Unk 2	11	11	10	10	12	11	10	10

The highest number of possible matches for each sensor pair with their corresponding window size is tabulated in table 4. This matching is not always true for all window sizes. For instance, in the case of sensor 1 *Unk 1*, the window size 50 had 10 matches, which represents that there are two mismatches between the *Sig* and *Unk 1* value. Again, the mismatch may arise from any of the materials, electrical, and/or external parameters discussed early in this section, where the level of contribution may be complex at these higher temperature measurements. At least one match for each temperature is required to showcase the working of a functional wireless sensor. If there is a proper correlation between the temperature signatures (*Sig*) and the unknowns (*Unk 1* and *Unk 2*) for at least one algorithm, then there will be a minimum of 6 matches. In the case of windows with 5 or fewer matches, both the cross-correlation and minimum absolute algorithms failed to match at least one temperature. Therefore, the window sizes with less than 6 matches can be ignored for further discussion.

Overall the total number of matches presented in table 4 shows the effectiveness of the signal processing method presented in this work. Generally, for all the sensor pairs, the window size ranging from 5 to 500 showed the best match. Furthermore, it can be inferred from table 4 that *Sensor 2* and *Sensor 3* showed a much better performance than the sensor 1. As mentioned earlier, this again proves the stability of the electrical contact to the exterior Pt lead wires played a vital role in the wireless sensor response. The algorithms were shown to be quite effective in matching the *Sig* with the *Unk 1* and *Unk 2* with respect to the window size. The signal processing method developed in this work can also accurately predict the match for each temperature with respect to their window size. This process allows the user to check if at least one algorithm matches each temperature.

Table 5 shows a summary table of the matching process for *Sensor 1* for both unknown measurements (*Unk 1* and *Unk 2*) using the two best window sizes shown for each. In table 5, AE represents the minimum absolute error and CC represents the cross-correlation algorithms. The checkmark (✓) infers a match and the cross (✗) infers a mismatch between the unknowns and the temperature signatures. For *Sensor 1-Unk 1* with window size 100, the table 5 shows that a perfect match for each temperature was achieved. Whereas, the window size 500 shows one mismatch for CC at 500°C and one for AE at 600°C. In total there are 10 matches and this summary shows that at least one algorithm satisfies for each temperature. Similar results observed for the *Unk 2* with a window size of 500, but with an exception there is a mismatch at 700°C. Both algorithms failed to match at 700°C, which means that the window size 500 cannot be used to characterize the wireless data. Similar data analysis was performed on the second and third sensor pair shown in fig. 14 to estimate the window(s) which showed at least one match for each temperature.

Table 5. Summary of the results of the signal processing methods using the split window technique for the two best matches for Sensor 1 using the spectrum shown in fig. 13.

Temperature	500°C		600°C		700°C		800°C		900°C		1000°C	
Algorithm	AE	CC	AE	CC	AE	CC	AE	CC	AE	CC	AE	CC
Unk 1 (WS 100)	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Unk 1 (WS 500)	✓	✗	✗	✓	✓	✓	✓	✓	✓	✓	✓	✓
Unk 2 (WS 10)	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Unk 2 (WS 500)	✓	✓	✓	✓	✗	✗	✓	✓	✗	✓	✓	✓

^{a)}WS: window size

The signal processing algorithms can accurately predict the temperature signatures, but it can also translate the data points to the frequency bandwidth which is more applicable in real-time characterization of the wireless response. The signal processing technique utilized in this work does not require complex Fourier analysis or other similar techniques to translate the data to the frequency domain. The translation of the data to the frequency domain was performed by equally splitting the frequency bandwidth (10 – 80 MHz) into 2400 individual data points. The frequency was then compared with the RSS (also contains 2400 data points) to translate the data into frequency domain. Table 6 shows that the frequency bandwidth for all three sensors with their respective window size. For simplicity, we chose the two best matching window sizes for the comparison between each sensor pairs. The frequency regions shown in table 6 represents the bandwidth where the algorithms have the best match for each temperature signatures. A larger bandwidth represents a well-pronounced matching window which spans over a larger data sets within the spectrum. In the case of *Sensor 1-Unk 1*, the window size 100 had a bandwidth of ~1.1 MHz, whereas the window size 500 had a bandwidth of ~3 MHz. Although both windows show similar matches, window 500 was preferred over window 100, because it spans over a larger bandwidth (~3 MHz). A similar result can be observed for the other two sensor pairs as well. *Sensor 2* has a frequency bandwidth of ~3 MHz, and *Sensor 3* has a bandwidth of ~3 - 4 MHz. A general trend from the above analysis shows that the active sensing frequency bandwidth lies from 40 – 65 MHz. This shows the effectiveness of the signal processing developed in

this work to accurately predict the temperature signatures irrespective of a predetermined computational model, which does not take into effect of the other extrinsic parameters on the sensor response. Additionally, the signal processing technique is adaptable to the change in the shape of the received signal.

Table 6. Frequency bandwidth of the two best matching windows with respect to each sensor pair.

Sensor Pair	Window Size	Frequency Bandwidth (MHz)
Sensor 1-Unk 1	100	44.06 - 45.15
	500	56.98 - 60.63
Sensor 1-Unk 2	10	48.35 - 48.53
	500	46.01 - 49.43
Sensor 2-Unk 1	1000	47.68 - 50.47
	2000	48.5 - 50.83
Sensor 2-Unk 2	100	62.27 - 65.15
	500	53.39 - 55.82
Sensor 3-Unk 1	500	53.89 - 55.86
	1000	40.51 - 44.88
Sensor 3-Unk 2	500	52.17 - 55.66
	1000	41.21 - 44.57

4. Conclusion

In this work, a robust passive wireless sensor was modelled, fabricated, and characterized for high temperature applications, where the active sensor/antenna design was composed solely of a conductive ceramic material. The initial composition consisted of a $\text{La}_2\text{NiO}_4/\text{Al}_2\text{O}_3$ particulate composite, which after extensive annealing to bond and stabilize the material, the composite reverted to a three phases mixture of LaAlO_3 , NiAl_2O_4 , and NiO . The sensors were patterned using the $\text{La}_2\text{NiO}_4/\text{Al}_2\text{O}_3$ particulate onto an alumina substrate using a two-step micro-casting process. This processing permitted the deposition of the particulate composite composed of micron-sized particles, which results in a relatively stable microstructure for the high-temperature

operation. At the same time, the micro-casting method showed the capability of printing the complex LC circuit at a micron-level resolution. The micro-casting process is an improvement over any physical vapor deposition method and direct ink method, where a composite ceramic thin film would be nearly impossible to deposit, and the films would generally display nanometer grain sizes that would be unstable over extended high temperature operations.

The wireless characterization of the sensors was performed between 500-1000°C with a frequency sweep between 10-80 MHz in an open-air muffle furnace without electrical insulation/shielding to mimic realistic working environment of the sensor. The sensors did not show the predicted peak shift at 500-1000°C near the 13.56 MHz frequency. The initial measurements indicated that many intrinsic and extrinsic variables may affect the sensor signature. This was unexpected since the sensors were fabricated in the exact same manner (using the same materials batches) and tested in the same exact manner. The seemingly scattered sensor response may have been exacerbated by the operation at the high temperatures used in this work, where this temperature range was not tested by previous researchers using a similar sensor design.

Most sensor developers would consider the sensor to be deemed unusable or a failure, since the expected signal shift was not identified for the given design at the various test temperatures. But after further review of the data, it was identified that the signal shifted from the centered frequency to other locations within the frequency bandwidth of the spectrum (depending upon the influences of the unidentified mechanisms and variables). It is believed that this phenomenon will be further seen in the future as the sensors are inserted in further “non-ideal” environments and at higher temperatures (which will translate into various influences on the electromagnetic, mechanical and chemical properties/behavior of the sensor). Therefore, an approach of analyzing the wider bandwidth of the spectrum was applied to characterize and monitor multiple peak shifts across the 10-80 MHz range. The work analyzed the received signal strength (RSS) instead of the S_{11}

parameter, or the phase angle ($^{\circ}$) shift, to characterize the wireless response of the sensor. The use of the RSS translates a temperature sensing problem to a signal processing problem, which allows for more than one degree of freedom for data analysis. Thus, the sensors reported in this work can respond to the temperature variation irrespective of its resonant frequency. A temperature signature database was collected by simultaneously measuring the wireless signal from the sensor and the temperature of the furnace with an external thermocouple. In consecutive measurements, the wireless response from the sensor was also collected without the external thermocouple and compared against the temperature signatures. Both the cross-correlation and minimum absolute error methods, when used in conjunction with the sliding window technique, achieved the best results with the sensors in matching the unknown temperature readings to that of temperature signatures. Although the shape of the RSS may vary between various sensor/antennae pairs, the sensors showed similar frequency bandwidth (40 - 65 MHz), where matches between the unknown reading with the temperature signatures were achieved effectively.

The signal processing methods proposed in this work were proven to be effective irrespective of the variation in the intensity and shape of the RSS. The robustness and adaptivity of the split-window technique proved to be very effective over conventional signal processing methods when the sensors operated in the harsh and varied conditions used in this work. The methods used were not only robust, but also adaptive to the change in the shape of the signal. As stated above, these signal processing strategies may be important in the further development of harsh-environment sensor systems where more complex sensor materials and operational environments are encountered in such extreme conditions.

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