Biodegradable Passive Resonance Sensor
Fabrication and Initial Testing

Timo Salpavaara[1], Ville Ellä[2], Minna Kellomäki[2] and Jukka Lekkala[1]
[1]*Department of Automation Science and Engineering, Tampere University of Technology, Korkeakoulunkatu 3, Tampere, Finland*
[2]*Department of Electronics and Communications Engineering, Tampere University of Technology, Tampere, Finland*

Keywords: Biodegradable, Passive Resonance Sensor.

Abstract: Biodegradable resonance circuits were studied. The circuits have a novel two-layer resonator structure without galvanic through hole vias. A patterned magnesium layers were evaporated on biodegradable PLA sheets by using a 3D printed mask. The circuits were assembled by heat sealing two magnesium patterned sheets together to encapsulate the circuit structure. An inductive link is used to wirelessly detect the resonance frequency of the circuit. The circuits were tested when immersed in de-ionised water and saline. According to the tests, the designed resonator structure can be measured in aqueous environment. The resonance of the tested circuit was observable at least for 51 hours. The concept still needs more development to extend degradation time and to increase the stability during immersion.

1 INTRODUCTION

Implanting sensors into the body of a patient is for fair reasons considered an extreme procedure regardless of the benefits that can be achieved. The main advance of the method is that the measurement is not hindered by the sensor-skin interface. The implanting procedure is more acceptable if it is done at an unavoidable surgical operation due to injury or disease. The threshold of using implantable sensors is even lower if sensors are not permanent and thus do not need a surgical removal operation. The biodegradable sensors are developed to utilize this niche.

One of the key features of an implantable sensor is to access it by using a wireless link. RF or inductive links are the most commonly used. The structure of a passive resonance sensor that utilizes an inductive link is very simple. Thus this method has been utilized in many implantable (Collins 1967; Chen et al. 2010) and biodegradable (Salpavaara et al. 2012; Luo et al. 2014) sensor studies. A passive resonance sensor is an LC circuit that is inductively read by another coil. In this configuration, the measurand is affecting the sensor coil or capacitor thus altering the measured impedance of the reader coil. Another approach is to link the measurand to the losses in the LC circuit. To create a biodegradable resonance circuit, coil and capacitor structures are needed. Thus the methods for making biodegradable patterned conductive and isolating layers are needed. In addition, the sensor has to be assembled and encapsulated without compromising the made structures.

Conductive parts or layers can be made of biodegradable metals (zinc, iron and magnesium) or conductive polymers. The conductive polymers, however, usually have too high resistivity for passive resonant sensor applications. One determining feature of the passive resonance circuit is the Q-value. In order to achieve high Q-values the resistance of the coil structure has to be small. For this reason good conductivity is needed and the fabrication method has to allow the preparation of thick conductor layers. Bounty et al. made conductive structures by using an electric discharge machining to pattern 3 mm think iron and magnesium sheets (Boutry et al. 2011). Luo et al. have used an alternative method which utilizes electroplating zinc and iron to create over 60 μm thick coil wires (Luo et al. 2014). The evaporation processes can be used to form patterned layers, however, the time needed to deposit thick layers is a problem. By using magnesium, high evaporation rates can be achieved. Magnesium has been used to form conductive areas to make wires, coils and

Salpavaara T., Ellä V., Kellomäki M. and Lekkala J..
Biodegradable Passive Resonance Sensor - Fabrication and Initial Testing.
DOI: 10.5220/0005255901270131
In *Proceedings of the International Conference on Biomedical Electronics and Devices* (BIODEVICES-2015), pages 127-131
ISBN: 978-989-758-071-0

capacitors (Hwang et al. 2012).

The possible substrate materials for biodegradable sensors can be polymers, silk and bioresorbable glass. Biodegradable polymers are divided into two groups: synthetic and those derived from natural resources. There are also two dominating degradation mechanisms: surface erosion or bulk erosion. The surface eroding polymers would make excellent candidates for sensor fabrication because their water absorption may be lower. One of the commonly used and well tested group of biodegradable polymer polylactides, (PLAs), unfortunately, degrades with bulk erosion. Other suitable candidates for biodegradable polymer substrates are polycaprolactone (PCL), polyglycolide PGA, poly(3-hydroxybutyrate) (PHB) and their co-polymers (Luo et al. 2014). Luo et al. used Poly (L-lactide) PLLA as substrate material for the passive resonance sensors. Another tested substrate material for biodegradable sensors is solution-casted silk (Hwang et al. 2012). They also proposed the use of MgO and SiO_2 as isolating materials which are needed to coat conductors.

Besides individual fabrication processes, an engineering problem arises with the assembly of the fabricated structures. In comparison to a typical silicon sensor fabrication, biodegradable structures are prone to get compromised when new processing steps are done over earlier layers. As an example, magnesium layers break if any following steps include water and many polymer structures will deform if they get in contact with solvents. To solve this problem, evaporation processes using physical masks can be utilized (Hwang et al. 2012). This method does not include photoresist masks which have to be patterned and then removed by solvent. Another method is to use embossing and lamination techniques (Luo et al. 2014).

In this paper, ongoing research for methods of designing and fabricating biodegradable passive resonance sensors is discussed. The methods of biodegradable sensor fabrication are developed and combined in a novel way to create a biodegradable resonator structure that can be modified to make implantable sensors. The designed prototype structure is assembled and tested in vitro.

2 FABRICATION METHODS AND DESIGN

The principal idea of the presented biodegradable resonator concept is to evaporate magnesium and silicon dioxide on PLA sheets through a physical mask. Then two sheets are stacked and combined to a resonator structure by heat sealing. This method encapsulates magnesium layers which are otherwise prone to water. Another leading design feature is to avoid galvanic through hole vias between the conductive layers. Instead, conductive layers on the separate sheets are connected by capacitors which are formed when the sheets are heat sealed together. The simplified cross-section of the design is shown in Fig 1.

The electrical schematic diagram of the designed circuit is presented in the Fig. 2a. This structure has four coil and two capacitor structures. The schematic does not include parasitic components. The measured electrical behaviour of the presented structure is similar to a simple RLC circuit and thus can be roughly modelled as shown in Fig. 2b. The resistance (Rs) is representing both the ohmic losses in the magnesium layers and the dielectric losses around and in the circuit. The inductor in the model (Ls) represents the combination of four circular planar coils (L1 to L4) on two opposing PLA sheets and the inductance of the other connecting wires in the circuit. In the final structure, four coils are placed in a way that a changing uniform magnetic field will induce currents that charge two main capacitances in the circuit. The capacitor (Cs) in the model is representing the capacitance of the two electrode areas that are located inside the circular coils and the combination of all parasitic capacitances in the structure. The Fig. 2b also illustrates the utilized wireless measurement principle.

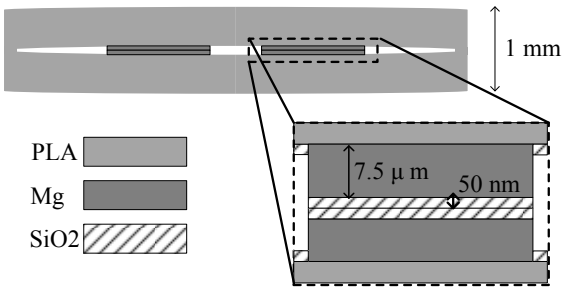

Figure 1: The cross-section of the resonator structure.

The idea behind this design can be derived from a concept where a planar coil and a parallel-plate capacitor are combined to an LC circuit by using a through hole via between the layers. Instead of via, another similar capacitor can be used to connect layers. The capacitors are in series which has to be taken account when the resonance characteristics are designed. This kind of design, however, would be unsymmetrical and in practice it would take almost similar area as two planar coils and two parallel-

plate capacitor electrodes per layer. Thus the set of four coils and two capacitors is adopted in this design to maximise the inductance in the circuit.

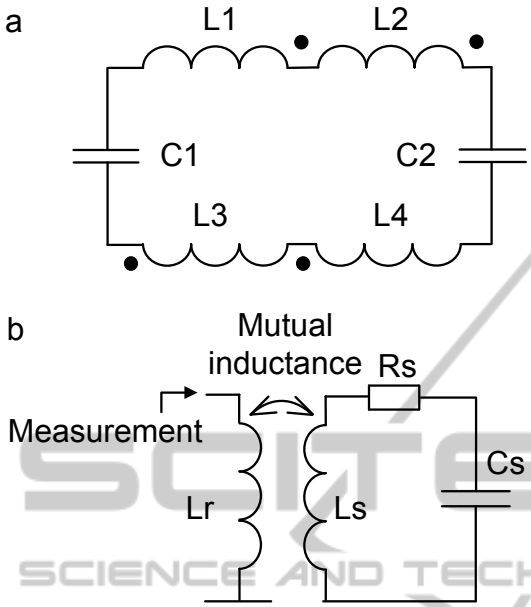

Figure 2: The electrical configuration of the fabricated resonance circuit (a). The measurement setup and the simplified model of the resonance circuit (b).

The substrate PLA sheets are made of PLA (Purac Corbion Purasorb PLD962). First, granules are melt processed to a 10 mm diameter rod. Then the rod is cut to smaller pieces which are compressed to 500 µm thick sheets by using elevated temperature and simultaneous pressure. One side of the polymer is compressed against glass sheet to make the surface of the polymer smooth enough for the evaporation process. Finally, the sheets are cut to 40 mm by 30 mm pieces.

The magnesium is patterned by using physical masks during the evaporation process. In this case, the masks were created by extrusion type 3D printing. This decision was made to test the capabilities of 3D printing and to accelerate the prototyping process. The extrusion path was manually designed to be as continuous as possible since the most of the irregularity of the 3D printed object occurs when the extrusion is started or stopped. Also one design criterion of the designed masks is that the objects have to support themselves. In practice this means that printed mask has to be continuous and there cannot be parts that sag when it hung on the roof of the evaporation chamber. The 99.99% pure magnesium was evaporated on the PLA sheets as a 7.5 µm ± 0.3 µm thick layer. The mask and the evaporated coil pattern are shown in Fig. 3. The magnesium layer was coated with 50 nm SiO_2-layer which acts as an electrical isolator between the capacitor plates.

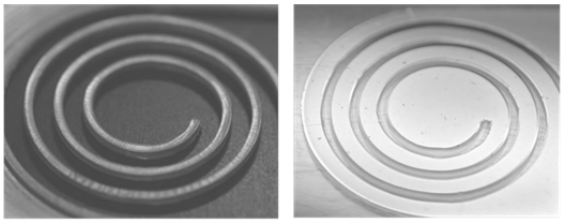

Figure 3: The 3D printed mask and corresponding magnesium pattern on PLA sheet.

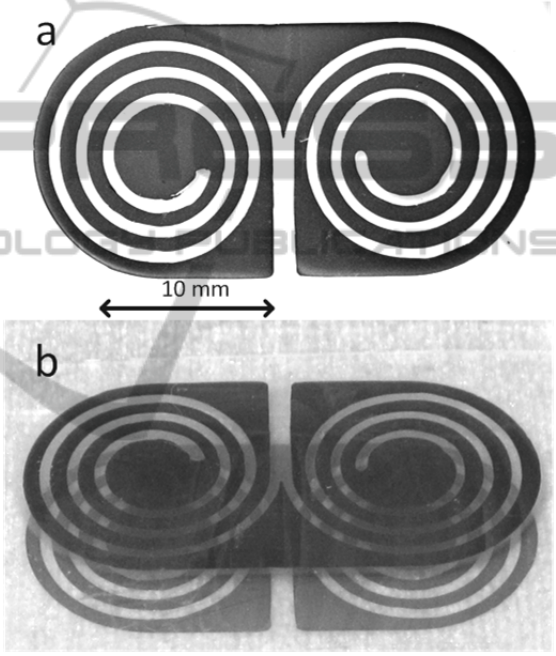

Figure 4: The magnesium patterned PLA sheet (a) and resonator structure assembly (b).

A resonator structure was assembled by stacking two patterned PLA sheets (Fig. 4) together with patterned sides facing each other. In addition, two patterned layers are placed in a way that coils on the opposite sheets turn opposite directions. The sheets were pressed together in a workbench and each edge was trimmed and heat sealed to waterproof the structure.

3 RESULTS

The impedance magnitude and phase of the resonators were measured in air by using an impedance analyser (Agilent 4396B with impedance

test adapter) and an external coil (diameter 40 mm). The impedance of the reader coil was also measured separately and this baseline was removed from the measurements. The two sample sensors have similarly shaped responses curves at clearly separate frequencies (Fig. 5). The frequencies of the phase dips (f_p) of these sensors are at 50.8 MHz and 58.9 MHz.

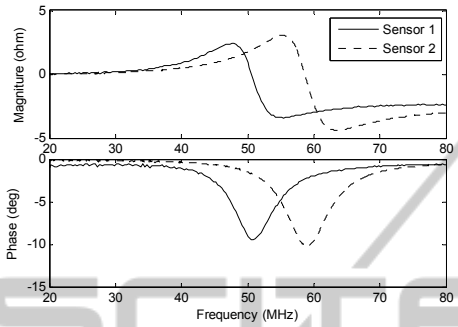

Figure 5: The magnitude and phase responses of the resonance sensors measured in air.

The sensor 1 was immersed in 30 ml of 9 mg / ml saline and sensor 2 in 30 ml of de-ionized water. The sensors were supported from the edges in a way that there was a 1 mm thick liquid layer under and over the tested object. The temperature of the test environment was 22 Cº. The changes of f_p are shown in Fig. 6. The initial drop due to the immersion is fairly similar in both cases. This is followed by the expected decrease of the f_p. After two hours of immersion, the f_p starts to increase again. This continues until roughly 6th hour in the case of sample one. After 6 hours, signals start to degrease again and in the case of sample 2, the decrease of f_p was faster. The measurement of the sample 2 was stopped after 8th hour. The f_p of sensor 1 increased dramatically between ten and twenty hours. The resonance was still clearly detectable until 51st hour.

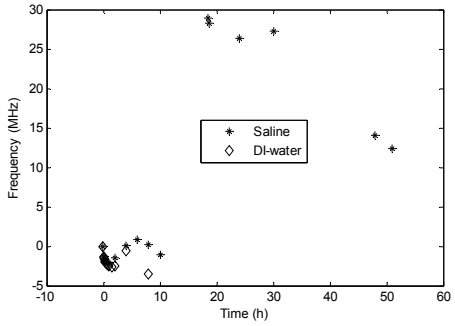

Figure 6: The frequencies of the phase dips of the tested sensors during immersion to de-ionized water and saline.

The degradation was also visually monitored (Fig. 7). There are no visual signs of degradation during the first 24 hours. The magnesium conductors start to show degradation after 48 hours of immersion to saline. After one week immersion in saline, the corrosion is clearly visible all over the magnesium patterns.

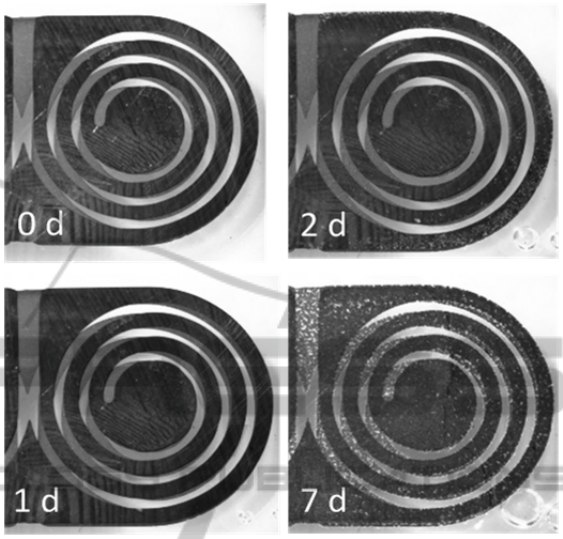

Figure 7: The degradation of the magnesium conductors begins to be visually detectable after 2 days immersion to saline.

4 DISCUSSIONS

The proposed structure for a biodegradable resonator has been successfully demonstrated. The scheme to avoid galvanic through hole vias can be realized. This simplifies the manufacturing process and the presented fabrication method has only a few process steps. The optimization of the presented structure may yield better results; however, modelling all the parasitic components in the structure is a complicated task.

The 3D printed physical masks worked well in this application. The performance of the printers will improve in coming years which may encourage using them for mask fabrication. However, it should be noticed that masks with fine details were only used to pattern magnesium in this study and their temperature limitations should be considered if used in other applications.

The general design is not especially sensitive to fabrication tolerances since the two made resonators have similar response curves. The difference in the resonance frequencies in air is not too significant considering manual assembly and the 3D printed masks. The main reason for the difference in the

resonance frequencies of this design is estimated to be the parallel plate capacitors assembly. In addition to alignment errors, the structure is sensitive to assembly pressure and the mechanical properties of the substrate PLA sheets. As process steps are developed, the resonance frequencies can set more accurately and variation will be smaller.

The main drawbacks of sample prototypes are the short durability in aqueous environment and the instability of the resonance characteristics due to the water absorption of PLA. The changes in the resonance characteristics of the tested circuits were so large that the measurements have to be verified with more parallel samples before any better conclusions can be drawn. Also the hydrolysis test of the device has to be longer and samples have to be measured with more dense intervals. It can be estimated that instability is caused by the absorbed water which changes the electrical fields inside the sensor structure. Other likely cause for instability is that unfixed capacitors plates will move as PLA absorbs water and swells. This may explain the large increase in the resonance frequency after first 10 hours of immersion.

The current study proved that the PLA sheets alone do not provide adequate encapsulation for magnesium. As a quick fix, magnesium oxide can be evaporated under and top of the magnesium layer since this material has been used to modify the degradation (Hwang et al. 2012). The magnesium can also be replaced with more slowly degrading metal like zinc, this, however, is unlikely to fix the instability problem and zinc has smaller conductivity compared with magnesium. Replacing PLA with a surface eroding polymer can be a valid option since the designed structure worked well in test conditions before water had time to absorb in the structure. Another option is to modify the water absorption of the PLA for example with a coating.

The structure is sensitive to pressure variations due to the mechanical changes in the assembled capacitors. This can be utilized in order to make pressure sensors or the sensitivity may be mitigated by filling the empty space that is left in the structure. Replacing the air in the structure with biodegradable hydrophobic material and fixing the capacitors plates together will make the structure more durable and may improve stability during immersion. In present design, only the edges of the PLA sheets were fixed.

The research will continue by modifying the designed resonator structure. The short-range goal is to develop a resonator that is stable for months. Then the design will be modified to be sensitive to a specific measurands like pressure or permittivity outside of circuit.

REFERENCES

Boutry, C.M., Chandrahalim, H. & Hierold, C., 2011. Characterization of RF Resonators Made of Biodegradable Materials for Biosensing Applications. *Procedia Engineering*, 25, pp.1529–1532.

Chen, P.-J. et al., 2010. Wireless Intraocular Pressure Sensing Using Microfabricated Minimally Invasive Flexible-Coiled LC Sensor Implant. *Journal of Microelectromechanical Systems*, 19(4), pp.721–734.

Collins, C.C., 1967. Miniature passive pressure transensor for implanting in the eye. *IEEE transactions on bio-medical engineering*, 14(2), pp.74–83.

Hwang, S.-W. et al., 2012. A physically transient form of silicon electronics. *Science (New York, N.Y.)*, 337(6102), pp.1640–4.

Luo, M. et al., 2014. A Microfabricated Wireless RF Pressure Sensor Made Completely of Biodegradable Materials. *Journal of Microelectromechanical Systems*, 23(1), pp.4–13.

Salpavaara, T. et al., 2012. Biodegradable encapsulation for inductively measured resonance circuit. In *Bioinformatics & Bioengineering (BIBE), 2012 IEEE 12th International Conference on*. Larnaca: IEEE, pp. 11–13.