Ink-jet printed ring resonator with integrated microfluidic components

Laura Jasińska
Department of Microsystems, Faculty of Microsystem Electronics and Photonics, Wrocław University of Science and Technology, Wrocław, Poland
Krzysztof Szostak
Department of Telecommunications and Teleinformatics, Faculty of Electronics, Wrocław University of Science and Technology, Wrocław, Poland
Milena Kiliszkiewicz
Department of Microsystems, Faculty of Microsystem Electronics and Photonics, Wrocław University of Science and Technology, Wrocław, Poland
Piotr Słobodzian
Department of Telecommunications and Teleinformatics, Faculty of Electronics, Wrocław University of Science and Technology, Wrocław, Poland, and
Karol Malecha
Department of Microsystems, Faculty of Microsystem Electronics and Photonics, Wrocław University of Science and Technology, Wrocław, Poland

Abstract

Purpose – The main purpose of this study is to test the performance of the ink-jet printed microwave resonant circuits on Low temperature co-fired ceramics (LTCC) substrates combined with microfluidic channels for sensor applications. Normally, conductive patterns are deposited on an LTCC substrate by means of the screen-printing technique, but in this paper applicability of ink-jet printing in connection with LTCC materials is demonstrated.

Design/methodology/approach – A simple microfluidic LTCC sensor based on the microstrip ring resonator was designed. It was assumed the micro-channel, located under the ring, was filled with a mixture of DI water and ethanol, and the operating frequency of the resonator was tuned to 2.4 GHz. The substrate was fabricated by standard LTCC process, and the pattern of the microstrip ring resonator was deposited over the substrate by means of an ink-jet printer. Performance of the sensor was assessed with the use of various volumetric concentrations of DI water and ethanol. Actual changes in concentration were detected by means of microwave measurements.

Findings – It can be concluded that ink-jet printing is a feasible technique for fast fabrication of micro-strip circuits on LTCC substrates, including microfluidic components. Further research needs to be conducted to improve the reliability, accuracy and performance of this technique.

Originality/value – The literature shows the use of ink-jet printing for producing various conductive patterns in different applications. However, the idea to replace the screen-printing with the ink-jet printing on LTCC substrates in connection with microwave-microfluidic applications is not widely studied. Some questions concerning accuracy and reliability of this technique are still open.

Keywords FEM, LTCC, Microwaves, Ink-jet printing, Microfluidics

Paper type Research paper

1. Introduction

Nowadays, many types of highly integrated microfluidic systems incorporate microwave circuits for rapid heating and dielectric parameters detection purposes. Such hybrid devices are relatively small and have many advantages, including fast response time, good resolution and real-time control over reaction parameters. A microwave circuit can act as a heating or sensing component. The first type of application is commonly used to control heating of the substance placed inside the

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microchannel, which is frequently used in chips dedicated to the PCR (Polymerase Chain Reaction) process (Marchiarullo et al., 2013; Morgan et al., 2013; Shah et al., 2010). The main advantage of this solution, in contrast to the resistance heating, is the precise heat delivery, almost exclusively, to the fluid located in a microchannel. The second field of applications includes testing/sensing some changes in the fluid parameters by means of microwave signal reflection and transmission. Within these types of systems, the biomedical applications such as a cancer cell detection (Grenier et al., 2010, 2013), the concentration of glucose (Kim et al., 2015; Schwerthoeffer et al., 2013), ethanol (Sohrabi et al., 2014) or humidity measurements (Bogner et al., 2017; Jones et al., 2017) can be mentioned.

In the literature, various techniques for developing the microfluidic-microwave microsystems can be found. One of the most common trends is creating a planar microwave circuit on a dedicated glass-epoxy laminate with dielectric parameters suitable for microwave applications. The next step is adding a microfluidic channel made from another (frequently polymer) material over the selected part of the microstrip circuit (Abduljabar et al., 2014; Chretiennot et al., 2013; Ebrahimi et al., 2014; Zarifi and Daneshmand, 2016). Another approach is the use of polymeric substrates, such as PDMS (polydimethylsiloxane), onto which the conductive paths are deposited using various techniques (Leroy et al., 2015; Shah et al., 2010). In contrast to the previously mentioned methods, the low temperature co-fired ceramics (LTCC) technology (Low-Temperature Co-fired Ceramics) allows fabricating the monolithic microfluidic devices with integrated microwave components on the same substrate material (Macioszczyk et al., 2017; Malecha et al., 2019).

Currently, screen printing is the most popular method of depositing a mosaic of conductive paths on the LTCC substrates. However, it can be a very time consuming and demanding technique (Drela et al., 2014; Słobodzian, 2015). Therefore, it would be good to have an alternative to screen printing, especially in the field of rapid prototyping. Ink-jet printing seems to be a promising solution. It was successfully used to make a microfluidic device with a microwave antenna on a paper substrate. In that case, similar to the glass-epoxy laminates, the microchannel was made of PDMS and then inserted under the paper layer with the antenna structure printed on it (Su et al., 2014). As a part of our earlier work, we used the ink-jet printing method to fabricate microstrip transmission lines (Szostak et al., 2019). Our studies have shown that the microwave parameters of the microstrip line deposited using the ink-jet printing are satisfactory for the rapid prototyping of microwave, conductive structures on LTCC substrates. On the basis of the obtained results, we decided to develop a microstrip ring resonator with an integrated microfluidic channel. At the first stage, we started with numerical modelling of the ring resonator integrated with a microfluidic channel.

2. Numerical model

2.1 Preliminary calculations

Dimensions of the microstrip ring resonator were roughly approximated using the following equations (Pozar, 2012) (for geometry):

$$r = \frac{nc}{2\pi f\sqrt{\varepsilon_{eff}}} \quad for \quad n = 1,\ 2,\ 3,\ \ldots \tag{1.1}$$

$$Z_0 = \frac{120\pi}{\sqrt{\varepsilon_{eff}}\left[\frac{W}{d} + 1.393 + 0.667\ln\left(\frac{W}{d} + 1.444\right)\right]} \tag{1.2}$$

where r *[m]* is the mean radius of the designed resonator, n is the mode number, f *[Hz]* is the resonant frequency, c *[m/s]* is the velocity of the light in the free space, ε_{eff} *[-]* is the effective permittivity of the substrate, W *[m]* is the microstrip line width, d *[m]* is the thickness of the substrate (which is equal to the distance between the microstrip line and the ground plane) and Z_0 *[Ω]* is the characteristic impedance of the microstrip line. As the resonator was aimed for applications connected with aqueous solutions, the resonant frequency of the ring resonator was chosen to be at 2.4 GHz. The microfluidic channel was placed underneath a part of the ring, and its shape was selected so to obtain high microwave response to changes in fluid parameters. In turn, the geometry of the feeding lines was designed to obtain the characteristic impedance of 50 Ω. Approximated parameters of the resonator derived from equations 1.1 and 1.2 gave us the basis for electromagnetic simulations and further geometry refinement.

2.2 Numerical modelling

The final geometry of the ring resonator was designed using a full-wave electromagnetic simulator, CST Microwave Studio. The resonant frequency (2.4 GHz) was adjusted under the assumption the microchannel is filled with deionized (DI) water with relative permittivity equal to 78. The proposed microfluidic sensor was modelled to measure the variations in ethanol concentration in water. The change in concentration was observed as a shift of the resonant curve in frequency due to changes in the dielectric constant of the measured solution. The dimensions of the ring resonator were optimized on account of the highest possible sensitivity.

The microwave circuit consists of two microstrip lines coupled with the resonator ring. The inner diameter of the ring is 13.5 mm, and the outer one is 15.5 mm. The width of the microstrip line is 1 mm and the length of the feed lines was 13.4 mm. The gap between the feed lines and the ring resonator was 0.1 mm. The simulated transmittance ($|S_{21}|$) of the resonator in the function of the relative permittivity of the dielectric material placed in the microchannel, as well as the response of the circuit at the resonant frequency, are presented in Figure 1a and 1b.

As we can see, the microwave circuit exhibits the highest value of $|S_{21}|$ for the dielectric material with relative permittivity equal to 78 (deionized water). When the dielectric constant of the liquid placed in the microchannel decreases, a shift in the resonant frequency (to the right) and some fluctuations in $|S_{21}|$ are observed. The proposed sensor, when tuned to the resonant frequency (2.4 GHz), shows a nearly linear change in $|S_{21}|$ in the function of the relative permittivity of the solution, with an R-square coefficient equal to 0.9987 (Figure 1b).

3. Materials and methods

As a substrate material, the LTCC Du Pont 951 with the green tape thickness of 254 μm and dielectric constant equal to 7.5 (Jasińska et al., 2019), was chosen. The substrate stack consists of four green tape layers, as illustrated in Figure 2.

Figure 1 The transmittance (|S21|) parameter in the function of the relative permittivity of the dielectric material placed in the microchannel (a) and the response of the resonator at the resonant frequency of 2.4 GHz (b)

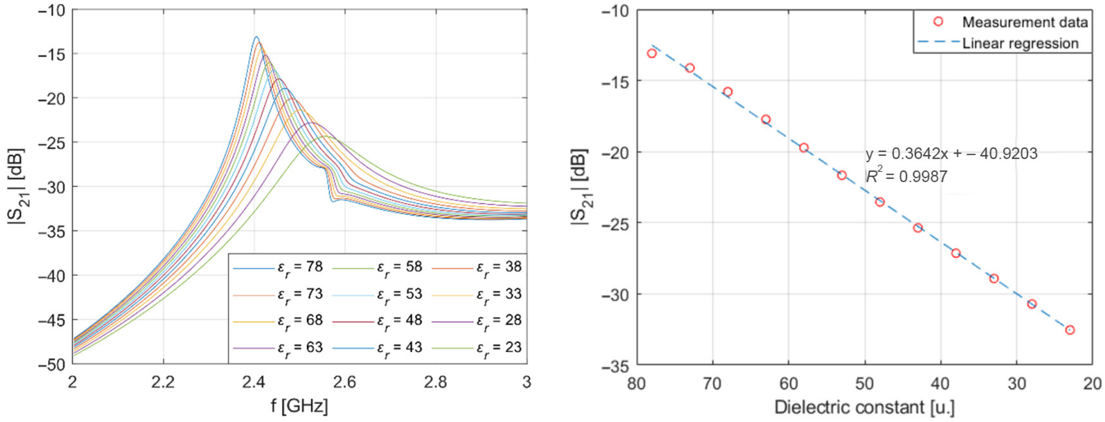
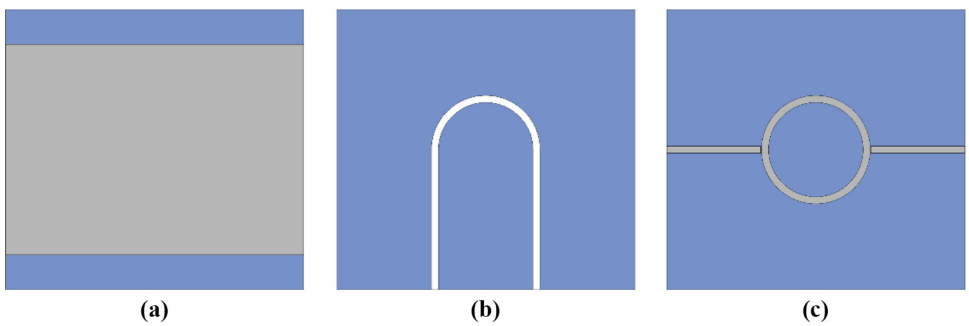


Figure 2 Schematics of the LTCC substrate stack layers (a-the first layer with the ground plane, b-the second and third layers with the microchannel, c-the fourth layer with the resonator geometry)



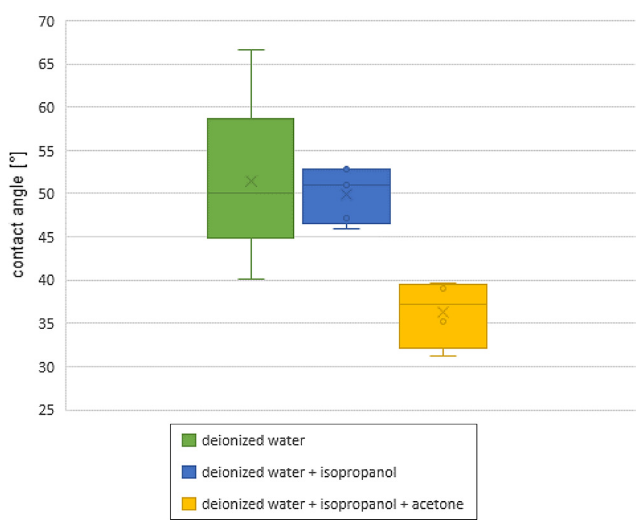
The microchannel was placed in two inner layers of the substrate (layers 2 and 3, Figure 2b). The microwave circuit was deposited on the upper layer of the substrate with the use of the ink-jet printer. The ground plane was deposited on the bottom of the module, and this time, because of cost savings, a fast version of the screen printing method was used (layer 1). Furthermore, each layer includes pads for the clamp edge-launch SMA connectors.

The first step of the technological process was cutting the shapes of the microchannels and the holes dedicated to the SMA connectors using the LPKF ProtoLaserU (Nd:YAG, λ = 355 nm). Then, the layers were stacked and laminated by the isostatic press for 10 min at a temperature of 70°C and at a reduced pressure of 5 MPa. The reduction of the pressure in the lamination process was imposed by the high risk of the microchannel deformation. Afterwards, the laminated structure was cofired with the standard temperature profile (with T_{max} = 880°C) in the chamber furnace Nabertherm THC 03/16.

The geometry of the microwave circuits was deposited on the cofired substrate with the use of the ink-jet printing technique. The overall printing process consists of the succeeding steps: substrate cleaning, pattern printing and sintering.

The accuracy of the ink-jet printing method and the uniformity of the deposited layer are highly sensitive to appropriate substrate preparation. Hence, in the first step, the contact angle in the function of the substrate cleaning method was analysed with the goniometer PG-X (Fibro System AB). The results are shown in Figure 3.

Figure 3 A boxplot with the contact angle as a function of different types of cleaning



As a result, the substrates were cleaning in the deionized water for 30 min in the ultrasonic washer Branson 8210 at room temperature. After drying, the substrates were back again placed in the ultrasonic washer in the isopropanol for 30 min at room temperature.

Afterwards, the microstrip circuit was deposited by the Dimatix DMP-2831 ink-jet printer with the use of ink with silver nanoparticles (AMEPOX Microelectronics Ltd. Nano Ink JP-60n) and 10 pL drop-volume cartridge. Printing parameters were chosen as follows: hot-plate temperature set at 40°C, 1 active nozzle, and 5 μm drop space. The consecutive pattern was then sintered at 240°C for 1 h in the Binder GmbH convection oven.

After the sintering process, the stainless steel needles, which acted as inlet and outlet of the microchannel were glued using the epoxy resin. Finally, the clamp edge-launch SMA connectors were attached. The fabricated resonator is presented in Figure 4a and the placement of the microchannel in the substrate was shown in the image obtained by CT scanning (see Figure 4b).

The usefulness and sensitivity of the fabricated resonator were verified experimentally. As a tested fluid, the water solutions with the various volumetric concentration of the ethanol (0 per cent, 10 per cent, 30 per cent, 50 per cent, 70 per cent and 90 per cent) were chosen. Prepared solutions were injected into the microchannel by the syringe pump Braun Perfusor Space. The 2-port measurements of the scattering parameters were then carried out using the VNA (Vector Network Analyzer) Rhode&Schwarz ZLT in the frequency range from 1 to 6 GHz. The used test-bench is shown in Figure 5.

4. Results

The measurements were performed for two resonators. The results for the first and second one are presented in Figures 6 and 7, respectively.

As it can be seen in Figure 6a, the resonant frequency of the first, printed resonator with the microchannel filled with the deionized water is 2.84 GHz, which is the value shifted by 0.44 GHz in comparison to the electromagnetic simulation. In Figure 7b, the $|S_{21}|$ parameter values for different ethanol concentrations at resonant frequency are presented. The obtained results indicate the good ring resonator linearity for

Figure 4 The picture of the fabricated ring resonator (a) and the CT scanning image of the microchannel (b)

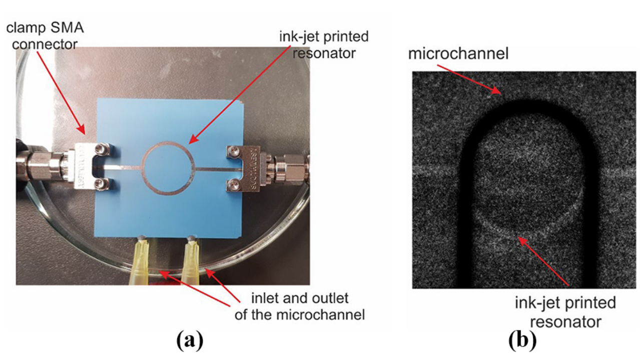
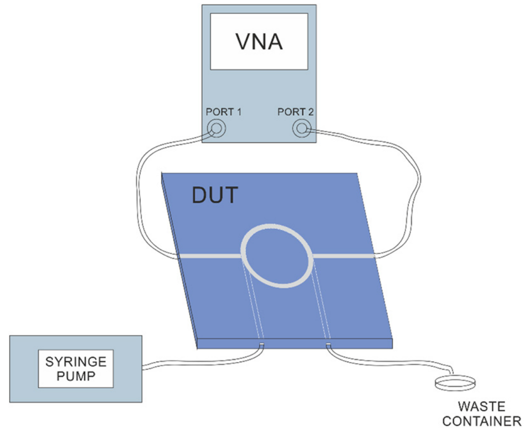


Figure 5 The schematic of the measurement stand (DUT-Device Under Test; VNA-Vector Network Analyzer)



low ethanol concentrations with the R-square coefficient of 0.957 and the mean sensitivity at the level of 0.96 dB per each 10 per cent of the additional volumetric concentration of ethanol. What is more, good detection/resolution properties were obtained for frequencies ranging from 2.75 to 2.84 GHz.

In the case of the second ring resonator, the resonant frequency achieve for the microchannel filled with the deionized water is 2.57 GHz (Figure 7a), which is the value shifted by 0.17 GHz in comparison to the simulation results obtained in CST Microwave Studio. The received value is much more consistent with the simulation than in the first resonator (Figure 6a). As can be seen in Figure 7b, the relationship between the volumetric ethanol concentration and the $|S_{21}|$ parameter at the resonant frequency is characterized by the poor linearity and worse sensitivity than in the previous case (Figure 7b) with the R-square coefficient of about 0.926 and the mean sensitivity at the level of 0.73 dB per each 10 per cent of the additional volumetric concentration of ethanol.

Furthermore, some microwave signal distortions can be observed in both cases, which is probably caused by the realization of the junction between the strip conductor and ground plane of the microstrip ring resonator feeding lines by an unfirm mounting of the clamp, edge-launch SMA connectors. Furthermore, the differences in the obtained resonant frequency for the first and second resonator can be probably caused by the ink-jet manufacturing inaccuracy beacuse to the ring-shape of the resonance circuit and to the application of the mentioned edge-launch SMA connectors. Moreover, inaccurate deposition of the metallization, that acts as the microwave resonant circuit (for example, the unequal distance between the feed lines and ring resonator for ports 1 and 2), can input the differences in the changes of the transmittance S_{21} in the function of the various ethanol concentrations.

5. Conclusions

In this article, the ink-jet printed microstrip ring resonator with the integrated microchannel has been designed and fabricated on the LTCC substrate. The investigated device was arranged to work at a resonant frequency of c.a. 2.4 GHz, due to the high

Figure 6 The transmittance (|S21|)of the first, ring resonator for the frequency range from 2.5 GHz to 3 GHz in a function of the various volumetric concentration of ethanol in deionized water (a) and the changes of the |S21| parameter for different concentrations of ethanol at f = 2.84 GHz (b)

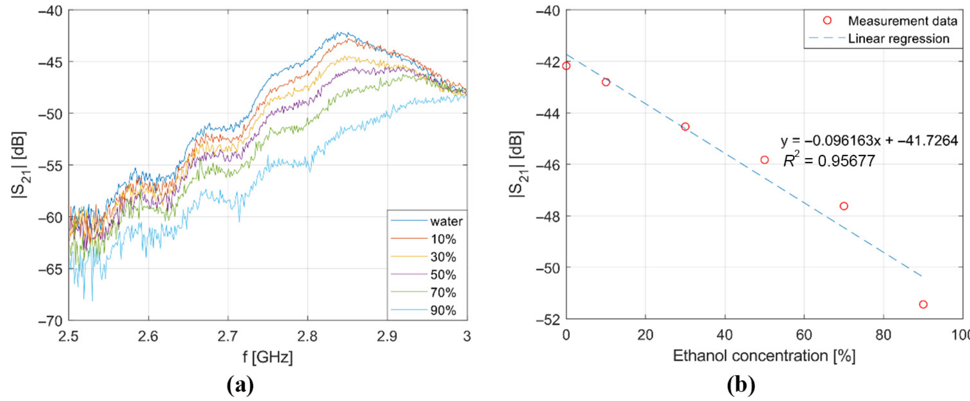
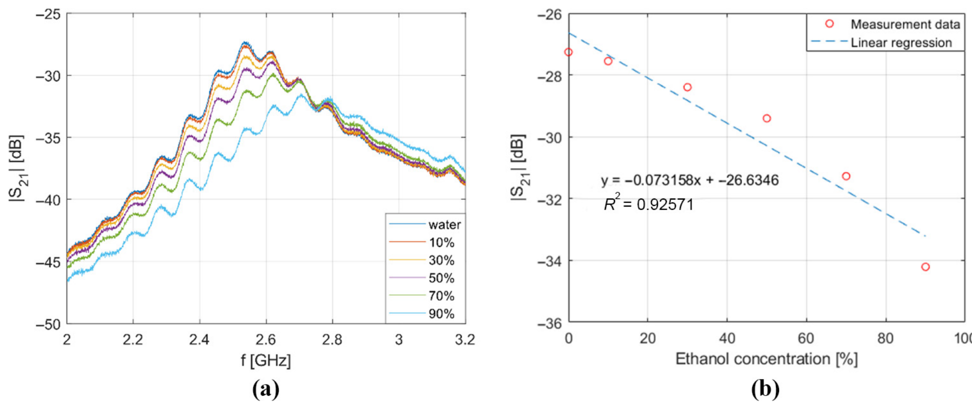


Figure 7 The transmittance (|S21|) of the second, ring resonator for the frequency range from 2 GHz to 3.2 GHz in a function of the various volumetric concentration of ethanol in deionized water (a) and the changes of the |S21| parameter for different concentrations of ethanol at f = 2.57 GHz (b)



dielectric losses of the DI water around this frequency. The main principle of operation of the described microwave sensor was the shift in resonance frequency for various liquids introduced to the microchannel. The solution of the different ethanol concentrations in deionized water was introduced as a test sample because of the high difference between the dielectric constant of DI water and ethanol.

The proposed microwave circuit geometry was designed using the CST Microwave Studio full-wave electromagnetic simulator and verified experimentally with the utilization of two ink-jet printed microstrip ring resonators. The first one of measured resonators was characterized by the linear relationship between the transmittance and the ethanol concentration in the DI water at the resonant frequency (for low concentrations). However, the obtained resonant frequency was equal to 2.84 GHz, which was 0.44 GHz higher than the designed one. In turn, the second resonator was defined by the resonant frequency equal to 2.57 GHz, which was 0.17 GHz higher than expected. Nevertheless, its sensitivity decreases for the volumetric ethanol concentration lower than 50 per cent. The significant variation between the designed and measured parameters was probably caused by the insufficient precision of the conductive strip deposition using the ink-jet printing method, high roughness of the LTCC substrate and unfirm connection of the clamp SMA connectors.

The ink-jet printing technology presented in this paper proved to be a useful tool in the field of the rapid prototyping that involves microwave circuits on the LTCC substrates. However, the necessity of identifying the ink-jet printing steps that can influence on the parameters of the printed microwave circuits appears. Probably, many process parameters can have an impact on the printed microwave circuit quality, such as the number of the printed layers, the chosen conductive ink and its rheology, the drop space, the hot plate temperature, the way of sintering and others. Because of the multiplicity of the factors, further research will be based on the Design of Experiment methods. Mentioned research will be conducted to achieve the improvement of the reliability, accuracy and performance of the printing the microwave circuit on the LTCC substrates by ink-jet technique.

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Corresponding author

Laura Jasińska can be contacted at: laura.jasinska@pwr.edu.pl