

# Portable and Precise Measurements with Interdigital Electrodes at Wide Range of Conductivity

Jaromir Hubalek, Radek Kuchta

Brno University of Technology, Dept. of Microelectronics  
Udolni 53, 60200 Brno, Czech Republic  
{hubalek, kuchtar}@feec.vutbr.cz

**Abstract.** A planar structure of the electrodes employed in conductivity measurements is utilizable at small range of conductivity measurement because of small sizes, planar ordering and technology which significantly influence the impedance behavior. The paper presents new approach in the field of precise electrolytic conductivity measurements with planar sensors. This novel measuring method was suggested for measurement using thick-film screen-printed interdigital electrodes (IDEs). Correction characteristics over a wide range of specific conductivities were determined from an interface impedance characterization of the IDEs. A local minimum of the imaginary part of the interface impedance is used for corrections to get linear responses. The method is seeking the optimal frequency for determination of specific conductivity of the solution by IDEs in order to achieve a highly accurate response. The method takes precise conductivity measurements in concentration ranges from  $10^{-6}$  M to 1 M without cell replacement.

**Keywords:** interdigital electrodes, accuracy, conductometry, thick-film sensors.

## 1 Introduction

The impedance spectroscopy is well known method to study interfaces solution-electrode. It is very important in corrosion science [1]. In the area of sensors it helps us understand as the ions reacts with the electrode, to determine their concentration. The ion membrane of biosensor can be characterized with this method as well [2]. Generally the impedance characterisation is tool to find the working conditions for conductometry. This knowledge increase significance when the electrode system is much distanced from ideal theory conditions where large macroelectrodes and high frequency for measurement are utilized because of the interface capacitance close to zero and only solution resistance (conductivity) measuring [1]. In a case of miniaturise electrodes as IDEs, additionally that are created in a planar form, the impedance spectroscopy at wide range of conditions has to be applied. The sizes, spherical currents, porosity or/and roughness and the edge effect can absolutely change impedance behaviour and make the accurate conductivity determination very difficult. A parasitic capacitance of the planar electrode structure with the substrate precludes high frequency measurements. A work of Sansen's team was focused on the conductivity error determination using comb-like structure [3]. Their results were

verified over a concentration range from 0.1 M to 0.2 M NaCl solution. They defined that the cell constant depends on the correction factor and a ratio of the electrode width and interelectrode distance. Optimal IDEs design was found to decrease error of measurement [4]. The double layers of microelectrodes, planar structure of IDEs, roughness significantly impact the total capacitance behaviour of the electrode interface impedance [5], [6], [7]. The impedance range can be suppressed at low and high frequencies which results to narrow band for the conductivity measurement. Therefore the method of precise conductivity measurement based on elimination of capacitance from measurements was discovered built-up on bipolar pulse technique [8]. However this technique also does not depress the dependence of the cell constant on IDEs structure at wide range of specific conductivities. The correction factor of our thick-film planar Pt IDEs strongly depends on measured conductivity [9]. The surface of the thick-film electrodes is very rough and porous which causes the electrode surface to be much larger than the thin-film equivalent. The sensitivity of the sensor can be higher in comparison with geometric sizes. Also, the capacity of the electrodes should be higher.

The electrode impedance theory described in [1], [10] can be compared with the interface impedance of the measured conductometric sensor. The interface impedance can be calculated from a cell constant determined by cross-sectional area  $A$  and interelectrode distance  $d$  over specific conductivity  $\kappa$  of the solution. Because geometric sizes do not represent the real cell constant, it is usually calibrated in a known solution.

The measurements took in local minimum of an imaginary part of the interface impedance correspond very well with measurement taken at high frequencies with large macroelectrodes [9]. In this point, at the ridge frequency, the capacitive part of the impedance interface is smallest and the resistance is approximately the sum of the solution resistance ( $R_S$ ) and charge transfer resistance ( $R_{CT}$ ). The  $R_{CT}$  is constant if the measuring voltage is smaller than 100 mV [11], but it depends proportionately on concentration as well as  $R_S$  [10]. Therefore, we can express that

$$R_S + R_{CT} \approx R_S = f(\kappa) . \quad (1)$$

A relative error of the interface impedance measurement can be determined at the minimum capacitive part by

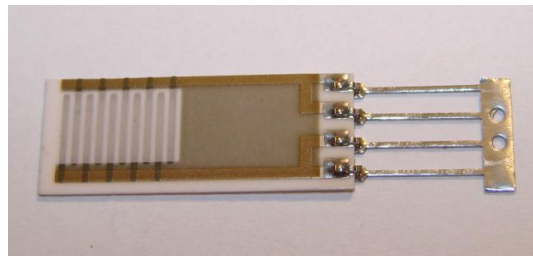
$$\Delta \equiv \left| \frac{Z_{measured} - R_S}{R_S} \right| = \frac{R_{CT}}{R_S} = \frac{1}{K_{cor(\kappa)}} . \quad (2)$$

$K_{cor(\kappa)}$  is a cell constant corrected for measured conductivity at the ridge frequency, where its value can be very close to constant. The parameter  $R_{CT}$  is factored into the  $K_{cor(\kappa)}$  according to (1).

## 2 Experimental

Thick-film interdigitated electrodes IDEs were fabricated from Pt paste (Dupont) screen-printed on alumina substrate and finally covered by over glaze paste. The

geometry of the sensors under test has had these parameters: width of fingers is 0.5 mm, length is 6.5 mm, interelectrode distance is 0.5 mm the electrodes are formed by 10 fingers. The impedance characterization was performed using Impedance analyser Agilent 4284. The sensing element is shown in Fig. 1. New instrumentation was designed, built-up on PCB and software based on our method that performs the measurement was created. The instrumentation was tested with the same sensing element.



**Fig. 1.** Thick-film screen printed sensor

The sensors were fabricated by deposition Pt thick-film comb structure with 5 fingers of every one electrode. Non-electrode parts were covered with overglass paste. An interface impedance characterisation of the thick-film sensor has been performed with an impedance analyser (Agilent 4284). The sensor, through testing, gives us better insight into sensor behavior.

### **3 Results and discussion**

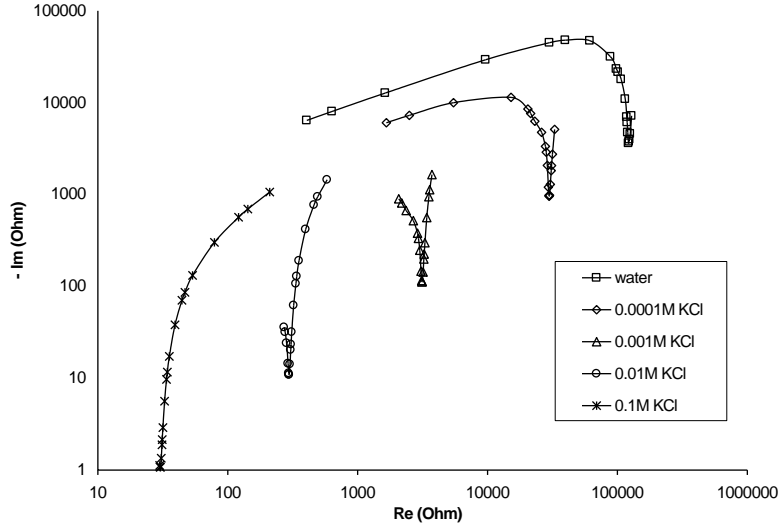
#### **3.1 Sensor characterization**

The frequency characteristics were found to depend strongly on the specific conductivity or solution concentration respectively. This dependence can be shown on logarithmic axis as plotted in Fig. 2.

Measured characteristics can be processed to a 3D plane graph as shown in Fig. 3. The measuring frequency and the conductivity specify the correction factor for the cell constant correction. It is clear that the frequency has to be changed as the conductivity is changed to get a very small dependence of the cell constant over a wide range of measurements.

Our experimental results confirm the behaviour, where only values at the ridge frequencies were taken within the minimum of the capacitance for the wide conductivity range of KCl solution [7]. The ridge frequency characteristic can be expressed in a complex plane as a power function of the real and the imaginary part of the interface impedance. The real part of the interface impedance has an

approximately linear relation with frequency and conductivity. Extracted relations are expressed in (3) and (4).



**Fig. 2.** Nyquist plots of impedance characteristics of the sensor at wide range of the conductivity

$$\text{Re} = C_1 \cdot f^{-\alpha} , \quad (3)$$

$$\text{Re} = C_2 \cdot \kappa^{-\beta} . \quad (4)$$

The fraction  $\alpha/\beta$  is approximately equal to 1. It is clear that if the real part of the impedance depends on the ridge frequency and conductivity, the relation between both variables can be derived. The frequency in the minima of the capacitance (the ridge frequency) was put into a resistance relation (5).

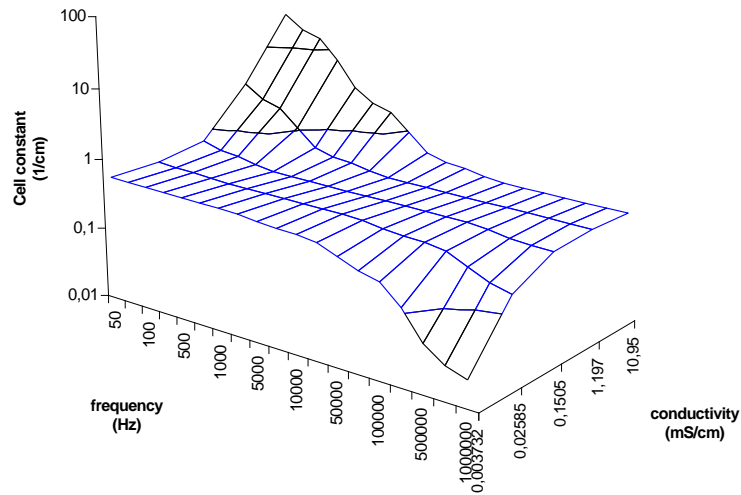
$$c_2 \cdot f^{-\alpha} = c_1 \cdot \kappa^{-\beta} . \quad (5)$$

Then the equation for calculation of measuring frequency at minima of the capacitance can be derived into (6). The new equation describes the relation between measuring frequency and specific conductivity. The relation is linear (Fig. 4).

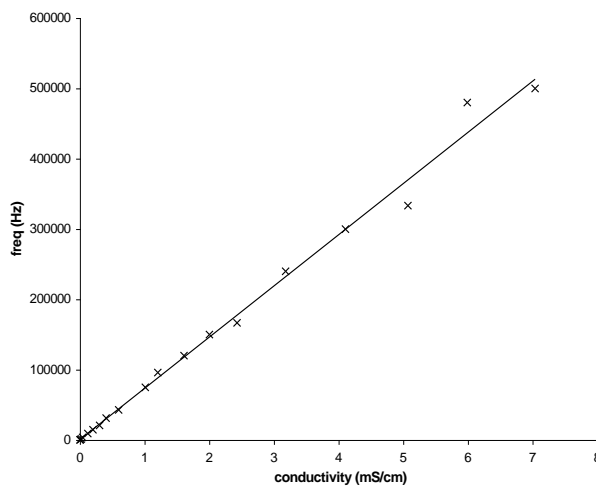
$$f = \left( \frac{c_1}{c_2} \right)^{-\tau} \kappa^{v/\tau} = c_C \cdot \kappa + c_{OFF} \quad (6)$$

The constants  $C_1, C_2$  have to be determined experimentally for each sensor. We found that the measurement has to be taken at the ridge frequency where the capacitance is minimal (Fig. 2) and real value of the impedance is invariable on a cell

constant dependency on the measured conductivity or concentration respectively and this real value very well proximate to the solution conductivity value.



**Fig. 3.** 3D characteristic of thick-film planar sensor



**Fig. 4.** Frequency dependence on measured conductivity at ridge frequency

As it was described above, the cell constant also depends on the measured conductivity. Because the interface impedance is measured, the cell constant should be derived from this impedance. An extracted equation is

$$K_{cell} = -c \cdot \text{Ln}(Z_{min}) + d \quad (7)$$

The constants  $c, d$  have to be determined experimentally. This correction equation can increase accuracy of the measurement. When the influence of the conductivity on the cell constant is very strong, the correction of the cell constant should be performed according to (6).

### 3.2 Suggested measuring method

Our novel method provides measurements exactly at the ridge frequency [9] corresponding to the measured conductivity value. The idea of measuring method appears from the cell constant dependency (see fig.4) where lowest change of the cell constant can be reached if the measuring frequency changes in respect of measured specific conductivity because of corresponding with the ridge frequency. We observed that it can be simple achieved with iterative algorithm. The responded conductivity is calculated from the cell constant corrected at this ridge frequency. Because the measured conductivity is not known at the start of the measurement, subsequent approximations to the ridge frequency and to the corresponding cell constant have to be performed in a few repeated measuring steps. This iterative method of the measurement can be demonstrated clearly in Fig. 5.

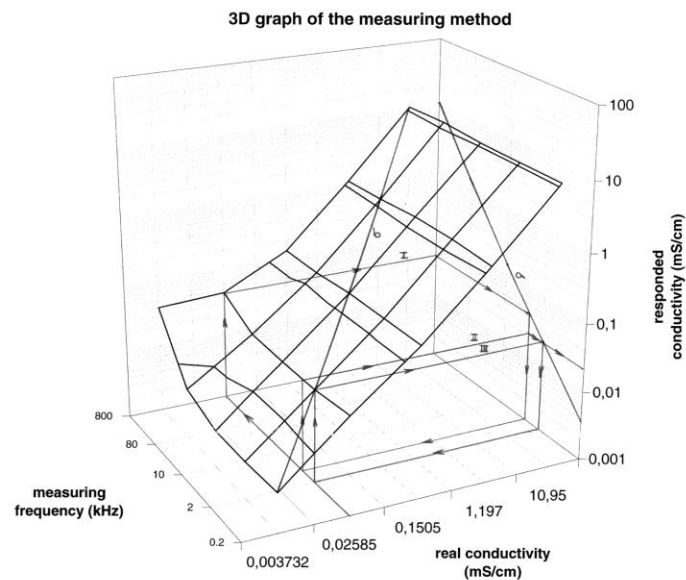
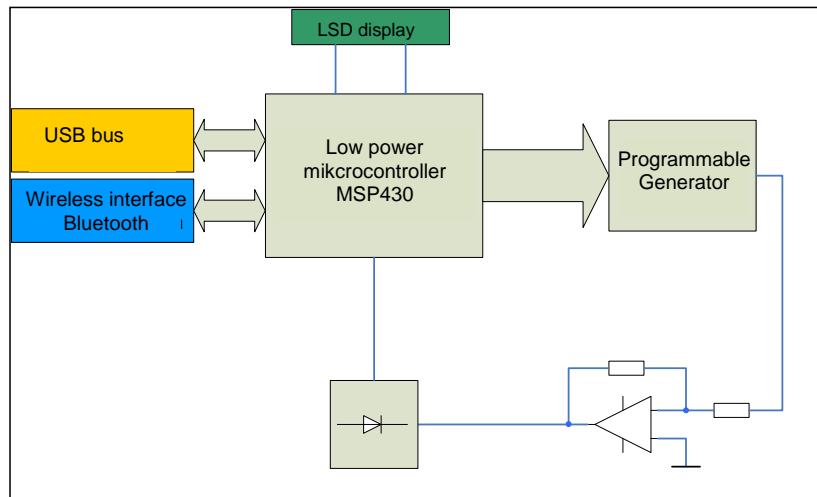


Fig. 5. Iterative steps of measurements

The 3D plot demonstrates the measured and the responded conductivity at different ridge frequencies and the corresponding cell constant of our planar sensor. If a solution with a conductivity value 0.02585 mS/cm is measured at the start frequency 800 kHz, the iteration steps go from front of x-axis to the back, where measuring

frequency is 800 kHz. Then the steps continue in direction of arrows to determine the responded conductivity. In the second step, a new measuring frequency is extrapolated from curve (a) to do the new measurement. This curve (b) is a 3D representation of (a). Steps are repeated until the responded conductivity is equal to the measured real conductivity (step III in Fig. 5). The method quickly finds the correct ridge frequency for a measured conductivity.



**Fig. 6.** Designed system providing method of automatic tuning of measuring frequency

This iterative method converges very quickly to the ridge frequency for the specific conductivity as show preliminary results in Tab.1. The measurements have done with Agilent impedance analyser, where the method was applied, and they helped us to determine maximal error to 2% at the range from 1  $\mu\text{S}/\text{cm}$  to 100  $\text{mS}/\text{cm}$ . This error was decreased below 1% by cell constant correction according to measured conductivity using (6). The responded conductivity is calculated from the cell constant corrected to the ridge frequency.

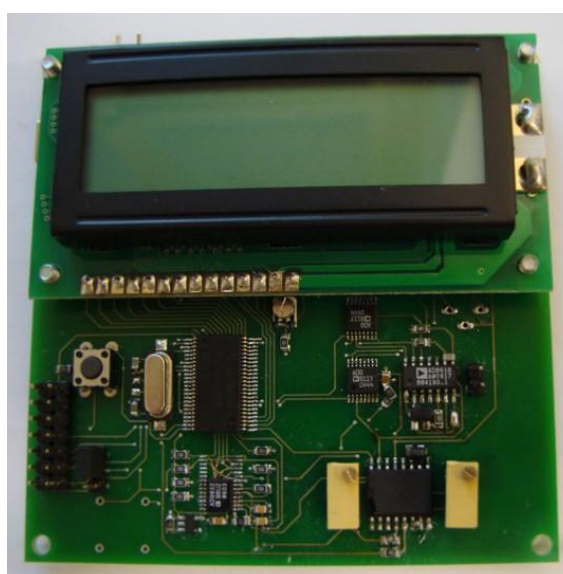
The method was implemented into new instrumentation. The microcontroller based system automatically providing the iterative method was designed. The block schematic is presented in Fig. 6. A fine tuneable oscillator connected with microcontroller including 16 bit A/D sigma-delta converter and analog circuitry including RMS to DC converter and low pass filter of 2<sup>nd</sup> order was utilized. All system is built-up as a portable battery supplied instrument. The system can be easily programmed through USB bus and data can be collected also by Bluetooth.

**Table 1.** Measured values during iterative steps of measurements

It.	Calcul. Freq (kHz)	Meas. Freq (kHz)	Re (kOhm)	Im (Ohm)	Conduct. (mS/cm)
1		1000	1.598	-589.2	0.1444
2	11.243	11.3636	2.1375	-77.81	0.1148
3	9.0871	8.9286	2.136	-77.03	0.1149
4	9.093	9.2307	2.128	-76.75	0.1154

5	9.1248	9.2307	2.128	-76.75	0.1154
It.	Calcul. Freq (Hz)	Meas. Freq (Hz)	Re (Ohm)	Im (Ohm)	Conduct. (mS/cm)
			500	235	-123
2	69132	68571.4	187	-6.25	1.3303
3	97569	96000	186.4	-5.94	1.339
4	98244	100000	185.9	-5.92	1.34
5	98491	100000	185.9	-5.92	1.34

The test instrumentation for precise specific conductivity performing our iterative method (see Fig. 7) was employed for comparison measurement with analyser Agilent. Our first results confirm validity of the method with accuracy 1% at wide



range of the specific conductivity without cell replacement.

**Fig. 7.** Final design of the instrumentation with display

## 4 Conclusion

The suggested method can improve the electrolytic conductivity measurement of the chemical sensor with planar screen-printed IDEs. Results show that a high accuracy of the measurement with the developed method can be achieved. The method was implemented into new designed instrumentation which performs the specific conductivity measurement automatically to reduce measuring time. The system can be connected to computer.

The elimination of the cell replacement with the maintenance of the accuracy over a wide range of the conductivity with our portable instrumentation using IDEs is the most important result of our work.



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