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Implementation of electrochemical impedance spectroscopy (EIS) for assessment of humidity induced failure mechanisms on PCBAs

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Abstract

Today's demands for steadily smaller and faster electronic devices lead to an ongoing miniaturization of electronics used in various fields. Current industry trends like e-mobility and autonomous driving are a significant motivation for this advancement. Reliability of electronics is an important aspect in all applications. In this context, particularly humidity induced failure mechanisms are of outmost importance. Temperature and humidity conditions greatly influence the durability of printed circuit board assemblies (PCBAs). The validation of humidity robustness is investigated using Surface Insulation Resistance (SIR) measurements that reveal electrochemical migration (ECM) and respectively dendrite growth. This method has several drawbacks due to its nature of direct current loading to show detailed information on water film formation and subsequent development of electrochemical failure modes. To enable the cause effect relationships of leakage current, ECM, and water film formation, Electrochemical Impedance Spectroscopy (EIS) is a promising method, since it will enable monitoring of water film formation on PCBA as a function of various parameters without perturbing electrodes due to corrosion. In this study, SIR comb structures of copper tracks on FR-4 substrates have been used for trials of a deeper understanding of EIS and a comparison with SIR measurements. The samples were exposed to climatic conditions, provoking a condensate formation on the surface. Measurements of SIR with an applied DC potential of 5 V and EIS measurements with an AC amplitude of 10 mV were conducted. The results from the EIS measurements formed the basis for electrical equivalent circuit modelling, portraying the reactions and the status of the PCB surface under changing climatic conditions and to investigate the impact of geometrical sample dimension on water film closing.

Keywords electrochemical impedance spectroscopy; surface insulation resistance; water film formation; electronic reliability; electrochemical migration

Introduction

Miniaturization efforts in technology, coming with new materials and manufacturing methods, impose durability demands that are difficult to reach. For the reliability of devices, humidity induced failures are one of the major problems, not seldom threatening the economic situation of manufacturers, for example due to early field failures. Electronics failure in humid conditions can often be attributed to condensation scenarios, leading to the formation of a thin water layer on the surface of a printed circuit board assembly (PCBA). With the water acting as electrolyte medium, various electrochemical processes are likely to occur. These involve manifold corrosive mechanisms causing intermittent functionality issues such as leakage current (LC) and electrochemical migration, along with other failure modes.

If due to an increased humidity level, the amount of water on a board is enough to bridge the gap between two oppositely biased metallic contacts, leak current failures and ECM are probable to occur. The dissolution of metal ions from the positively biased contact, transport of the ions via the water layer and their reduction at the negatively biased contact can lead to the formation of a metal dendrite. This eventually formed short circuit between the two contacts can result in a device failure.

The high complexity of PCBAs in terms of design and layout, material combinations, manufacturing technology and respectively residues, integration level as well as the varying operational environment conditions hamper a general understanding of humidity induced failure modes and finding a possibility to evaluate the humidity robustness of specific setups.

The formation of the water layer, its thickness and electrical properties can be regarded as determining step in the failure occurrence, may it be corrosion, dendrite formation or a similar process [1]. Commonly used direct current (DC) methods like SIR measurements to assess the humidity robustness of PCBAs, come with several drawbacks like high expenditure of time or destruction of the sample under investigation. Alternating current (AC) measurements have been investigated in order to obtain information on corrosive processes in various fields of industry and research [3, 4]. Electrochemical impedance spectroscopy is one kind of AC technique (schematically represented in Figure 1), that is based on the application of an AC signal $E(t)$ with a small amplitude ΔE to remain in a quasi-equilibrium stage while sweeping along frequency range over several orders of magnitude (ωt). The complex impedance Z of the system under investigation transfers the input signal to an output response, that can be displayed as a current $I(t)$ as a function of the exciting variables, phase shifted by a value ϕ and divided by the absolute value of the impedance $|Z|$.

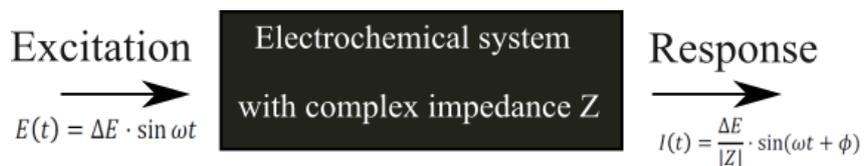


Figure 1: Schematic representation of the methodology of an EIS measurement.

In the context of electronics reliability, EIS methods have been used previously among others to monitor the corrosion rate of metals under thin electrolyte layers [5, 6], to study the interaction of flux residues with water vapor [7] and to investigate the impact of weak organic acids in flux systems [8]. Compared with DC measurements, EIS as AC method comes with the advantage of being non-destructive and it is able to distinguish between different capacitive and resistive processes and states [9]. This is achieved by using a broad frequency spectrum, whereas different conductive processes at the metal-electrolyte interface or respectively the electrolyte as conductive medium can be distinguished. This way, the measurement provides a system response to a given temperature and humidity that displays the system kinetics, which are masked by electrode polarization effects in the case of using

DC measurements. The electrochemical processes can be translated into electrical equivalent circuits, including components like resistors (R), capacitors (C) or imperfect capacitors like the constant phase element (CPE). Obtaining an electrical equivalent circuit that fits to recorded data and is congruent with the theoretical understanding of a system can provide an insight into the various electrochemical processes taking place. In this work, the EIS measurement in simultaneous implementation with SIR measurements, is aiming to gain a deeper understanding of the water film formation between conducting tracks of a PCBA. For this purpose, condensation tests on test boards have been conducted and the obtained data has been analyzed with a focus on the evolution of equivalent circuit parameters and the impact of geometrical sample dimensions e.g. different gap sizes.

Materials and Methods

Sample preparation

Both the SIR and EIS measurements were conducted using copper comb patterns of respectively 21 interdigital traces on glass-fiber reinforced epoxy resin (FR-4) of 1.6 mm thickness as substrate material. The manufacturing of the copper traces was executed by established photolithographic and subsequent wet etching processes. One test PCB was of the size of 125 mm x 125 mm and included four different comb structures, having the same copper width of 200 μm , but gap sizes of 100 μm , 200 μm , 300 μm and 600 μm (Figure 2).

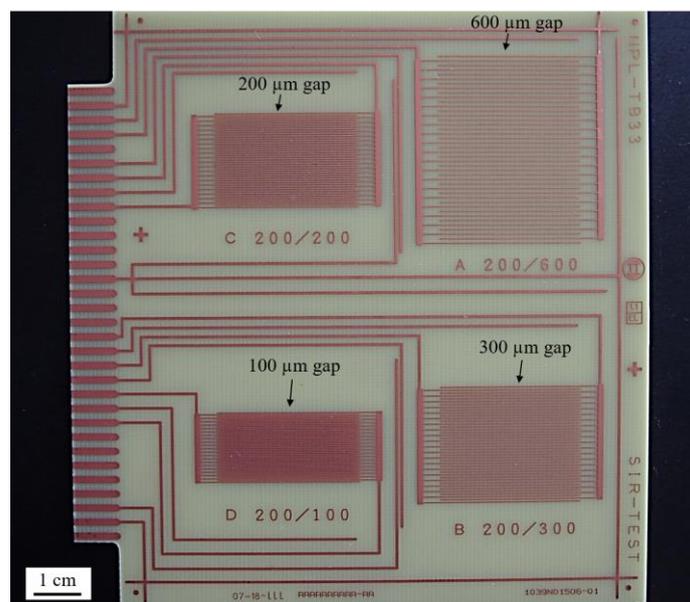


Figure 2: FR-4 sample board with copper comb structures of different gap sizes used for testing. Identical boards have been used for SIR and EIS testing simultaneously, whereas for both methods the 100 μm and 300 μm gap structures were used respectively.

Prior to testing, the boards were thoroughly rinsed with ultrapure Millipore water (specific resistance: 18.2 M Ωcm at 25 $^{\circ}\text{C}$) and isopropanol for one minute each. Subsequently they were blow-dried with compressed air and stored for at least 12 h in a dessicator filled with silica gel as drying agent to remove the remaining humidity from the PCBs.

Measurement setup

The temperature and humidity tests were carried out in a climatic chamber (PL-3KPH, ESPEC Corp., Japan), providing a temperature range between -40 $^{\circ}\text{C}$ and 100 $^{\circ}\text{C}$ with ± 0.3 $^{\circ}\text{C}$ accuracy and relative humidities (RH) between 20 % and 98 % with ± 2.5 % accuracy.

A Peltier cooling element (CP-031, TE Technology, Inc., USA) has been used to provoke condensation on the board surfaces by temperature reduction. For this purpose, the test boards were mounted onto the Peltier stage with a thermal paste (Silicone Heat Transfer Compound, Electrolube, United Kingdom). Additionally, a thermistor (MP-3193, TE Technology, Inc., USA) has been fixed onto a bare board part with thermal paste and thermal tape to monitor the actual temperature of the samples.

For the purpose of executing EIS and SIR measurements simultaneously, an electrochemical workstation (VSP, BioLogic Science Instruments, France) was used. The workstation possesses a total of five channels, two of which were used for EIS and two for contemporaneous SIR measurement for the 100 μm and 300 μm gap comb structures of the depicted samples (Figure 2). Both measurement types were conducted in the two-electrode setup.

SIR measurements were conducted with a DC voltage of 5 V. For the EIS measurements, an AC voltage with an amplitude of 10 mV was set, the frequency was scanned from 100 kHz to 100 mHz with 6 measurement points per decade and respectively 5 measures averaged for each of them.

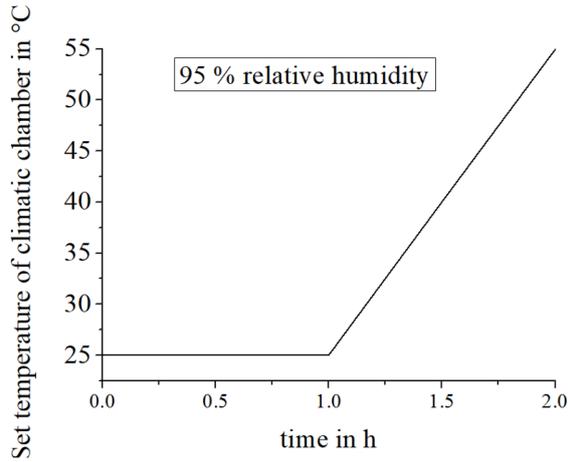
Experimental procedures

Two different kinds of experiments, that simulated operating conditions of automotive electronics and provoked condensation on the board surfaces, were conducted.

For Experiment 1, the climatic chamber without Peltier Stage was used, instead the two test boards for EIS and simultaneous SIR measurement were pasted on an aluminium block with the thermal paste. The climatic conditions they were exposed to involved a period of one hour at a constant climate of 25 °C and 95 %RH to achieve a steady state and have the samples' temperature adapted to the ambient temperature. The condensation was achieved by a subsequent chamber temperature increase to 55 °C within one hour, (heating rate of 0.5 °C/min), whereas the temperature difference between boards and ambient leads to condensate formation. (Figure 3a)).

In Experiment 2, condensation was achieved by local temperature reduction on the board surfaces with the Peltier stage. The two samples for EIS and SIR measurement pasted on the Peltier Stage were exposed to one hour of a chamber climate of 25 °C and 95 %RH, while the temperature of the board surface was measured using the thermistor. At the end of the equilibration period, the temperature of the Peltier Stage was reduced by 0.2 °C every nineteen minutes (Figure 3b)), while the chamber temperature was held constant at 25 °C. SIR and EIS analysis were started simultaneously after one hour of equilibration.

a) Experiment 1: condensation via chamber conditions



b) Experiment 2: condensation via Peltier Stage

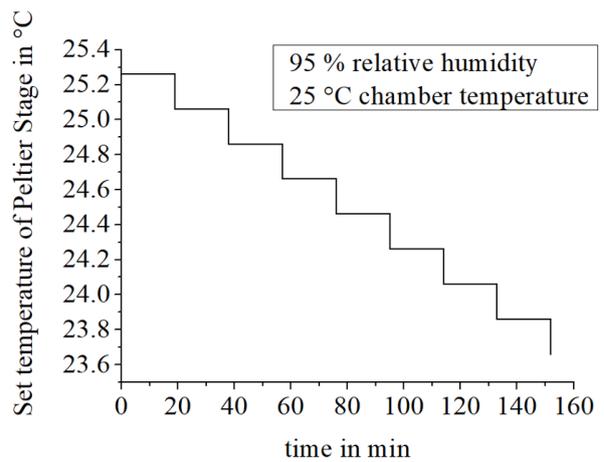


Figure 3 : Temperature profiles for experiment 1 with only the climate chamber (a) and respectively experiment 2 with the Peltier stage (b).

Results and Discussion

Comparison of SIR and EIS measurement

For both experiments, the SIR measurement provides the LC recording of the 100 μm gap and the 300 μm gap comb structures, while the EIS measurement yields the magnitude of the impedance and the phase shift of the respective two comb structures of the EIS samples.

The results of experiment 1 are depicted in the form of leakage current (Figure 4, black) from the SIR measurement as well as impedance and phase (Figure 4, red and blue) at a frequency of 100 kHz from the EIS measurement. The illustrated graph is based on the recorded values for the 100 μm gap comb structures, starting at 1.0 h, when the chamber temperature is increased after the equilibration period (cf. Figure 3a)).

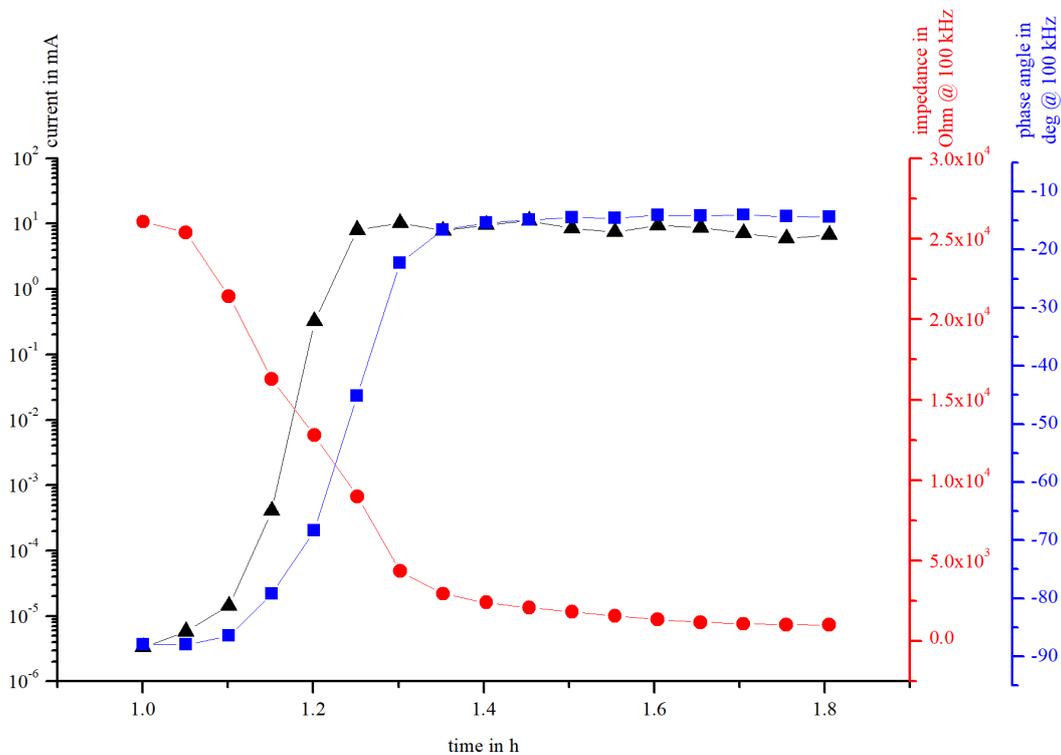


Figure 4 : LC values of SIR measurement and impedance and phase values at 100 kHz of the EIS measurement over the course of the condensation process of experiment 1.

The EIS measurement shows a decrease in modulus of impedance at 100 kHz from 26 k Ω to 6 k Ω within the first 25 minutes of the condensation process. The phase angle is simultaneously shifted from -90° to -15° . Both of the curve progressions point out the condensation process. The adsorbing water layer acts as conductive medium, decreasing the interdigital impedance between the comb structures and transferring the phase angle from capacitive characteristics to a resistive behaviour.

An increase of LC over 6 orders of magnitude displays the condensation process in the SIR measurement. After about 1.28 hours, a current in the two-digit mA-range is measured, indicating that water is bridging the gaps and that dendrite growth is enabled.

Examining the boards after the measurement confirms this, as it is shown in the microscope image in Figure 5. Figure 5a) shows a close up view of the 100 μm gap comb structures of EIS sample, which is unaffected in terms of short circuit formation due to the AC signal being insufficient for dendrite growth. The SIR sample (Figure 5b)) shows dendrite formation over several of the gaps.

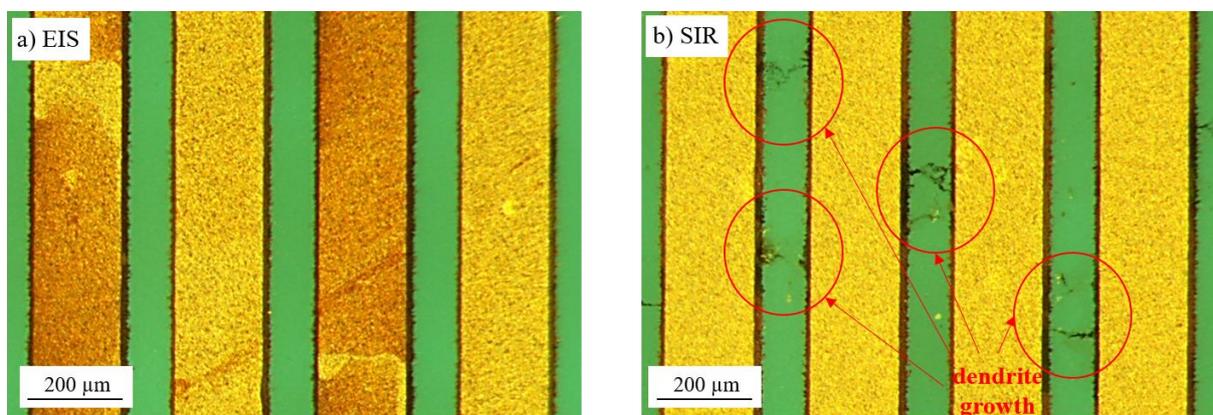


Figure 5 : Detailed images of the 100 μm gap comb structures that was subjected to a) EIS measurement and b) SIR measurement after experiment 1. Dendrite growth for the SIR-sample visible, the EIS sample is unharmed.

The results for experiment 2, where condensation is reached with active cooling of the boards using the Peltier stage are displayed in Figure 6, also being retrieved from the 100 μm gap comb structure.

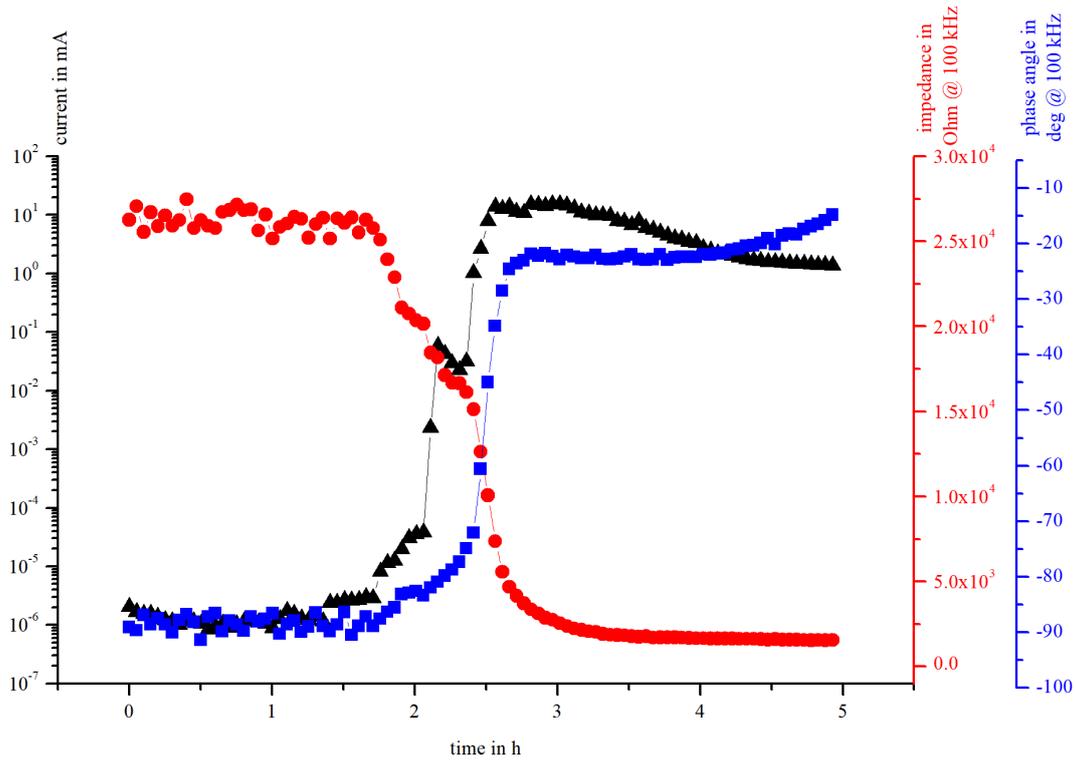


Figure 6 : LC values of SIR measurement and impedance and phase values at 100 kHz of the EIS measurement over the course of the condensation process of experiment 2.

The EIS measurement shows an impedance decrease from 26 k Ω to 3 k Ω along with a phase angle shifting from -90° to -14° . The water layer formation on the board surfaces is simultaneously depicted in the increase of the SIR-current, reaching mA values that point to dendrite formation.

Equivalent Circuit Fitting

The obtained complex impedance values from the EIS measurement of experiment 2 were used to perform electrical equivalent circuit modelling by usage of the software *ZSimpWin*. For fitting, the period of time from 2.5 hours to 3.2 hours (see Figure 6), where water adsorption had already started and continued until film formation, was used. The equivalent circuit that was the basis for the fit is shown in Figure 7.

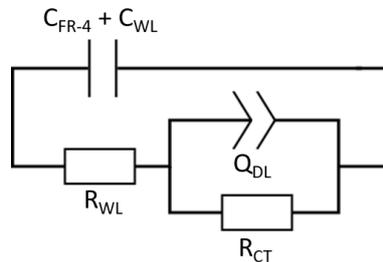


Figure 7: Electrical equivalent circuit used for fitting of EIS signal during water film formation in experiment 2.

The electrical equivalent components used are $C_{FR-4} + C_{WL}$, R_{WL} , Q_{DL} and R_{CT} . $C_{FR-4} + C_{WL}$ represents the capacitance of the FR-4 bulk between the comb structures and the capacitance of the adsorbed water, whereas the resistance of the water layer is characterized by R_{WL} . Q_{DL} is a constant phase element, incorporating the imperfection of the double layer

capacity by allowing a partly resistive behavior and R_{CT} , describing the charge transfer resistance at the copper – electrolyte interface.

Fitting was done for five subsequent measurements during the process of water film formation (goodness of fits: $X^2 \leq 1.051e-2$). In Table 1, the values obtained for R_{WL} , $C_{FR-4} + C_{WL}$, the exponent a_{DL} , that describes the capacitive/resistive character of the CPE ($0 \leq a_{DL} \leq 1$, $a_{DL} = 1$ for capacitance, $a_{DL} = 0$ for resistance) and R_{CT} are listed to show the impact of the water layer formation.

Table 1 : Fitting values obtained for equivalent circuit components during the course of condensation in experiment 2.

Time during experiment	R_{WL} in $k\Omega$	$C_{FR-4} + C_{WL}$ in pF	R_{CT} in $k\Omega$	a_{DL}
2.5 h	34.90	0.1127	231.4	0.4034
2.7 h	15.95	0.1139	119.6	0.4745
2.9 h	9.214	0.1234	84.43	0.4929
3.1 h	6.491	0.1340	64.88	0.5229
3.3 h	5.207	0.1396	58.86	0.5357

The resistance of the water layer R_{WL} decreases about one order of magnitude during the course of water adsorption. This can be attributed to the connection of the water droplets building up on the board surface and providing a connective pathway between the interdigital Cu tracks. Likewise, the capacitance is slightly increased, also hinting to more water adsorption and a correspondingly higher permittivity compared to only air.

The charge transfer resistance R_{CT} is similar to R_{WL} falling almost about one order of magnitude in the regarded condensation interval. This decrease is likely to be enabled by an increased water layer thickness, facilitating the charge transfer from metal to electrolyte.

Over the course of condensation, the CPE's exponent a_{DL} is slightly increased from 0.4 to about 0.54. Still the increase is not significant in a way that enables one to assume the CPE as a perfect double layer capacitor. It needs to be considered, that the CPE is a helpful component for modeling, but its functionality can not be transferred to the real-life system in the same convenient manner as it can be done for R or C elements. The usage of CPEs hampers the already existing ambiguity of an EIS signal in terms of potential electrical equivalent circuits. The fact that different arrangements of elements can be used for fitting, impedes a clear allocation of these elements and the underlying physical and chemical processes.

To avoid the usage of CPEs and obtain equivalent circuits that can be used for describing different states of the condensation process, further experiments need to be conducted. Based on more measurement data for a specific sample setup, it can be aimed at setting values for the framework elements like C_{FR-4} . This way, the focus can be put more on the actual water film formation between the conducting tracks and the problem of unambiguous assignments of equivalent circuits is restricted.

Influence of gap size

To illustrate the impact of the gap size (100 μm versus 300 μm) on the time-resolved formation of the water layer and its closing, the obtained phase angle at 100 kHz from the EIS

measurement has been regarded for both condensation experiments. The evaluation of the phase angles over condensation time is shown in Figure 8, starting at a point where the comb structures of both gap sizes do not yet show a water adsorption that is as significant as to be visible in the EIS signal (phase angle at -90°).

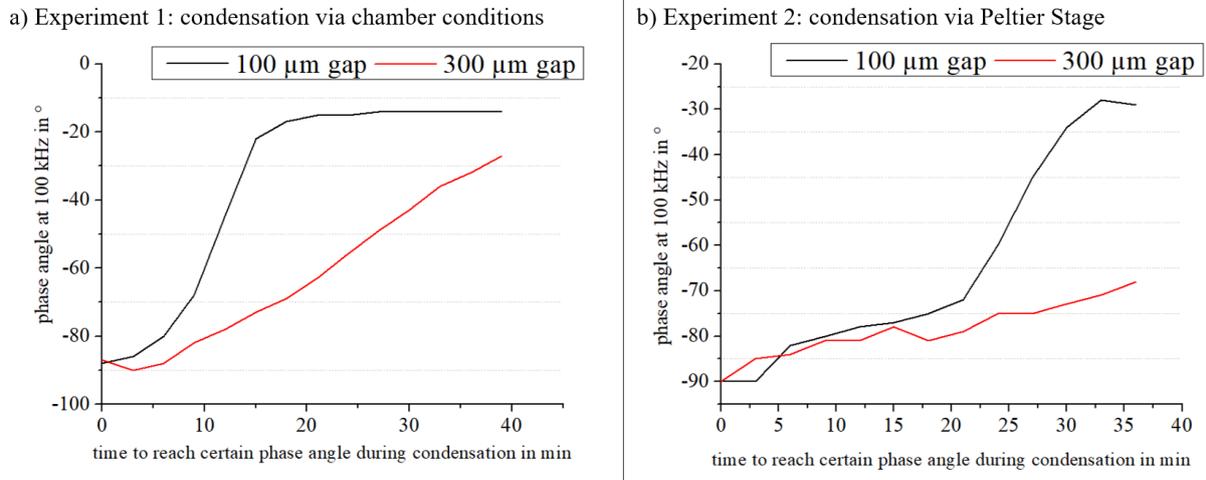


Figure 8 : Phase angle at a frequency of 100 kHz as a function of time during condensation process for comb structure with 100 μm and 300 μm gap size for experiment 1 (a) and experiment 2 (b).

As condensation progresses, for both experiments the phase angles for the 100 μm as well as the 300 μm structures shift to resistive values, indicating the water film formation. For experiment 1 (Figure 8a)), the 100 μm gap structure takes about 18 minutes time to shift the phase angle from capacitive (about -90°) to resistive (about -14°) values. Comparing with the measured phase angle for the 300 μm gap structure, it can be told that the experiment time was not long enough to achieve the same phase shift of -14° . Still it can be seen that after 40 minutes within the condensation process, the phase angle for the 300 μm gap structure has obtained a value of -27° , whereas the 100 μm structure has reached this value after 14 minutes, meaning a time delay of 26 minutes in this case.

Looking at the data of experiment 2 (Figure 8b)), where condensation was achieved more slowly due to a lower $\Delta T/\Delta t$, the influence of the gap size on closing time for the water film can be broadly seen as well. For the reason of the restricted measurement time, the phase angle for the 300 μm gap structure only reaches a value of -68° after 36 minutes, the 100 μm structure has obtained this value already after 22 minutes.

The results from both experiments go with the anticipated process of water film formation. First, small isolated droplets appear on the surface, they grow with ongoing condensation until adjoining droplets merge into larger ones and partially connecting starting to bridge the gap between the copper traces. The probability of droplets merging in a way that the whole gap is bridged decreases with increasing gap size, which is why the 300 μm gap closes slower than the 100 μm gap. This gap size effect is expressed in the different slopes $\Delta\phi/\Delta t$ within the graphs for experiment 1 and experiment 2 in Figure 8.

Comparing the graphs of experiment 1 and experiment 2 with each other, it can be seen that for experiment 1, the 100 μm gap structure shows a smooth transition of phase angle values from -90° to -10° . While for experiment 2, the phase angle first changes to -72° over the course of 21 minutes into the condensation and then changes its slope so that the shift towards -29° happens faster, within a time span of 15 minutes. Looking at the 300 μm structures, both experiments show a steady linear increase in phase angle with time, without a change in slope. In case of experiment 2, the measurement time might have been too short to potentially also show a bend in phase over time curve for the 300 μm structure like it is visible for the

100 μm structure. If such a change in $\Delta\phi/\Delta t$ takes place and how this reflects in water film formation in terms of different steps with different time constants for condensation processes based on different $\Delta T/\Delta t$ needs further investigation.

Conclusion

Comparison of EIS and SIR signals over the course of condensation show, that a $T\&RH$ condition that forms a water layer pronounced enough, to enable dendrite growth in the DC case, corresponds to specific EIS values. For the given test boards and temperature profiles, a phase shift of about -15° and impedance in the single digit $\text{k}\Omega$ area at a frequency of 100 kHz in the EIS measurement can be translated to dendrite formation in an SIR test.

An electrical equivalent circuit could be established, that described the electrochemical processes during water film formation in theory and also fitted the obtained EIS data. An increase in water layer thickness and consequently dendrite formation probability can be recognized by an R_{CT} and R_{WL} decrease as well as an increase in $C_{FR-4} + C_{WL}$. The significance of absolute values is limited due to usage of the CPE.

The influence of the gap size on the time-resolved closing of the water film could be shown by means of the phase angle evaluation. Dependent on the condensation condition, the larger gaps need longer time to be bridged by water. First results suggest that there is not necessarily a linear relationship of gap size and closing time over the whole course of the condensation period.

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