Analysis of Electrochemical Migration Kinetics Using Electrochemical Impedance Spectroscopy

Xiaofei He[1], Michael H. Azarian[1], Chunsheng Wang[2], Michael G. Pecht[1,3]
[1]Center for Advanced Life Cycle Engineering (CALCE)
University of Maryland
College Park, MD 20742 USA
Phone: +1-301-405-5323, FAX: +1-301-314-9269
[2]Department of Chemical and Biomolecular Engineering
University of Maryland
College Park, MD 20742 USA
[3]Prognostics and Health Management Center
City University of Hong Kong, Kowloon, Hong Kong

Abstract

The propensity of printed circuit boards to electrochemical migration has been assessed traditionally by using surface insulation resistance technique with a DC bias on standard comb structures. Different from this, an AC impedance measurement has been used to evaluate the kinetics of electrochemical migration process and provide detailed information about cell parameters such as solution resistance, charge transfer resistance, and double layer charging capacitance during dendritic growth. The solution resistance and charge transfer resistance decreased during the dendritic growth, while the double layer charging capacitance showed an oscillating nature. The dramatic changes of these parameters due to dendritic growth may be used as forerunner signals prior to dendritic growth and developed as a prognosis technique. A physicochemical model was fitted into the experimental results and a simulation was conducted. The simulation results confirmed the experimental data.

1. Introduction

Electrochemical migration (ECM) is the growth of conductive metallic structures on a printed circuit board (PCB) through an electrolyte solution under a DC voltage bias [1]. The ECM process consists of the following sequence of steps: path formation, electrodissolution, ion transport, electrodeposition, and filament growth [2]. Path formation is the creation of a favored path—a medium consisting of an electrolyte layer for metal ions to migrate. When a PCB is located in a humid environment, this step takes time. But if a PCB is directly located in a solution such as deionized water, this step can be omitted. Electrodissolution involves the oxidation of metals to generate cations at the anode (positive electrode). These metal cations can migrate under the influence of electromotive forces to the cathode (ion transport) and deposit at the cathode as neutral metal. As more and more neutral metal deposits on the nuclei (electrodeposition), a dendritic structure may grow from cathode to anode (filament growth). During its growth, the dendritic structure can produce a reduction in surface insulation resistance (SIR) between adjacent conductors, leading to current leakage and intermittent or catastrophic circuit failures.

Surface insulation resistance (SIR) measurement is a traditional means to determine the susceptibility of printed circuit boards (PCBs) to ECM and the degree of metal migration prior to the failure of PCBs, but it cannot differentiate the individual electrochemical processes contributing to ECM and identify the dominant ones at different dendritic growth stages. Identification of the rate limiting step in this process is essential for developing an accurate model of the time-to-failure, and helpful for devising strategies to eliminate or mitigate ECM. Despite its significance, there have been few published studies that analyze the kinetics of this failure mechanism in terms of its constituent parameters.

Electrochemical impedance spectroscopy (EIS) is popularly used in electrochemistry to study the electrode-solution interfaces and cell components. The reason why it can identify cell components is because the cell components, such as resistances, capacitances or inductances, have different frequency dependencies. The operation of EIS involves sweeping an AC signal (voltage or current) over a wide range of frequencies and measuring the responses (current or voltage) of an electrochemical system, thus providing detailed information on the constituent electrochemical components. The response has the same frequency as the input signal, but may have different amplitude and a phase-shift. The ratio between input voltages and current responses is defined as the impedance, shown as a complex value.

The impedances are always represented in Nyquist plot, Bode plot, and phase-shift plot. Nyquist plot is a plot with the real value versus the conjugate imaginary value of a complex number, as shown in Figure 1. Bode plot shows the magnitude of impedance versus frequency, always in a log scale, shown in Figure 2. Phase-shift plot shows the phase shift dependence on applied frequencies, shown in Figure 3. A more detailed description about the resistive, capacitive, and inductive circuits and their AC impedance spectra are given in Reference [3].

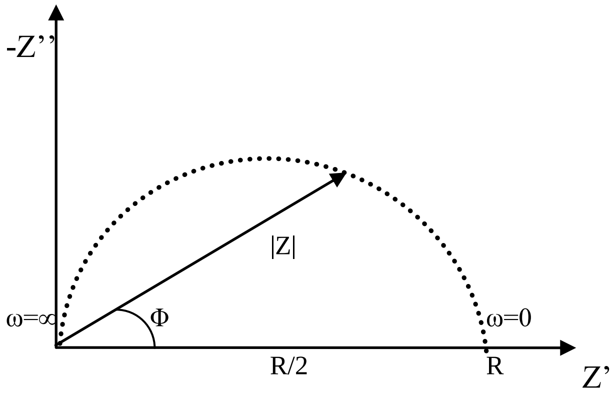

Figure 1: A typical Nyquist plot for an R-C parallel circuit.

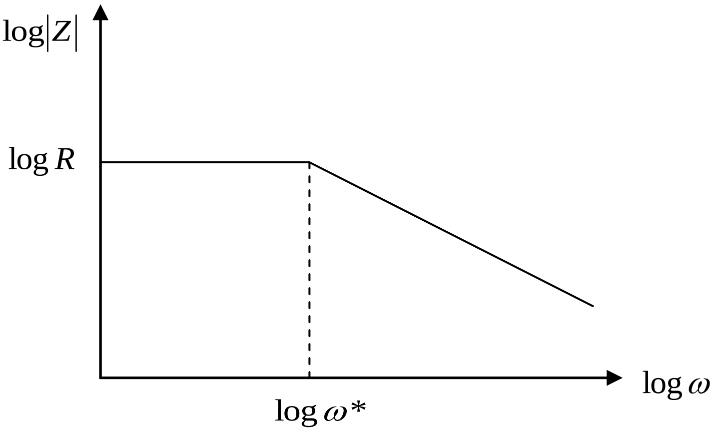

Figure 2: A typical Bode plot for an R-C parallel circuit.

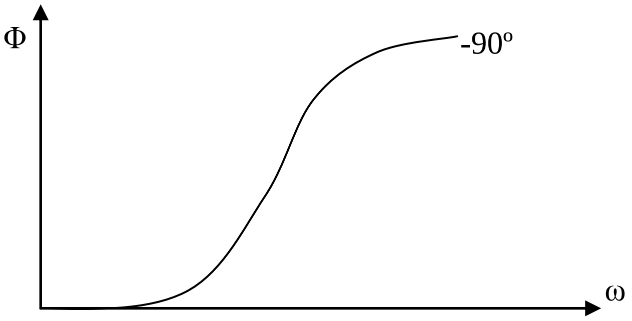

Figure 3: A typical phase shift plot for an R-C parallel circuit.

There are two ways to interpret EIS data. One is to start from a physicochemical model about the kinetics of an electrochemical process and seek to find a mathematical expression for the impedance [4]. Then the theoretical impedance calculations can be compared with experimental data to check the effectiveness of the physicochemical model. Another approach is to represent an electrochemical process with equivalent electrical circuits [3][5] and then use simulations or experiments to confirm the correctness of the representation. This approach is simple, but it can be ambiguous in some cases since more than one equivalent circuit may represent the same electrochemical process. In this study, the second approach is used. The standard PCB test comb structures used for testing ECM propensity can be prone to an ECM process, shown in Figure 4 and represented by a popularly used equivalent circuit, shown in Figure 5.

Figure 4: The electrochemical migration processes occurred on the surface of comb structure.

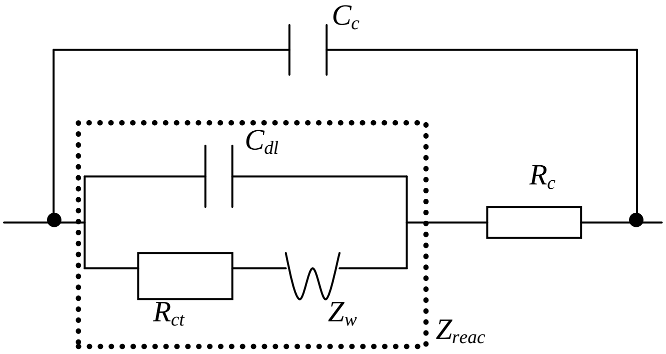

Figure 5: The equivalent circuit corresponding to Figure 4. C_c is electrical capacitance of the electrodes, C_{dl} is the double layer charging capacitance, R_c is the ionic transport resistance, R_{ct} is the charge transfer resistance, and Z_w is the Warburg diffusion resistance.

This paper presents EIS measurements of PCBs under bulk DI water conditions leading to dendritic growth. The constituents of the electrochemical system, such as charge transfer resistance, ionic transport resistance, electrodes capacitance, double layer capacitance, were determined using EIS. Optical microscopy was employed to record the dendritic growth after EIS measurement. A simulation was conducted to confirm the correctness of the equivalent circuit (Figure 5) used to represent the ECM process.

2. Experimental Setup and Procedure

Comb structures from standard test boards IPC-B-25 were used to grow dendrites and conduct impedance measurement. Since this comb structure contains many pairs of anode-cathode cells, dendrites can grow in many gaps. In order to control the growth and facilitate optical observation, only one pair of anode-cathode cell is maintained by severing the rest of the electrodes, as shown in Figure 6. So dendrites can only grow within one gap. Four samples were used to conduct the test, and they showed similar results. In order to monitor the system potentials thermodynamically, a micro-reference electrode Ag/AgCl was used and located close to the electrodes, as shown in Figure 6.

Deionized water with a resistivity larger than 18.2 MOhms·cm was used as the electrolyte to grow dendrites. The DI water was exposed to room ambient. Compared to humidity environment, bulk deionized water can directly create a migration path for ECM. Because the major objective is to study the kinetics of cell parameters during dendritic growth, one can avoid the time consuming path formation step when the comb is under humidity environment.

The impedance measurement system was Solartron analytical system. The frequency of AC signals ranged from 0.1 Hz to 100,000 Hz, which takes 2 minutes to finish one impedance measurement. A lower frequency (0.01 Hz~0.001 Hz) is required to detect the existence of diffusion, but that will take a few hours to finish one impedance measurement. Since the dendrites grow normally fast in DI water (a few minutes), a lower frequency cannot be used in this study.

Before starting the test, the comb structure was rinsed in DI water for 5 minutes, and then it was inserted into the electrolyte. The open circuit potential (OCP, the equilibrium potential of the whole system when the external current is zero) of bare copper electrodes in DI water was measured to be 0.05 V~0.08 V relative to the Ag/AgCl reference electrode. In order to record the system impedance baseline, a 5 mV AC signal relative to OCP was applied and the impedance was measured.

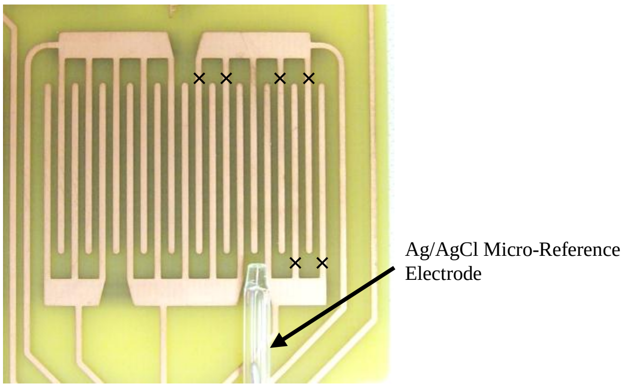

Figure 6: Comb structure and Ag/AgCl micro-reference electrode

A series of DC voltages (1.5 V~2.2 V) were applied across the anode and the reference electrode to allow dendrites to initiate and grow. The voltage across cathode and the reference electrode would be lower than that across anode and the reference electrode. So the voltage across the anode and cathode ranged from 3 V to 4.4 V. Each DC voltage lasted not more than 1 minute. If apparent dendritic growth was detected, the DC voltage would be stopped less than 1 minute. If no apparent dendritic growth was detected, the DC voltage would last for 1 minute. This approach could guarantee that the dendrites did not grow too fast and touch the anode. After each DC voltage application, a 5 mV AC signal relative to OCP was applied and the impedance was measured. So the DC and AC signals were applied in a cyclic fashion, to drive dendritic growth and to measure impedance respectively, until the dendrites finally touched the anode.

3. Results and Discussion

3.1 Kinetics of Cell Parameters during Dendritic Growth

Before starting the experiment, the baseline of the Nyquist plot was recorded by applying 5 mV AC signal relative to OCP across the cell, as shown in Figure 7. The frequency ranges from 0.1 Hz to 100,000 Hz. The DC potential was 0 V relative to open circuit potential (OCP) to guarantee there was no driving force for dendritic growth during the impedance measurement.

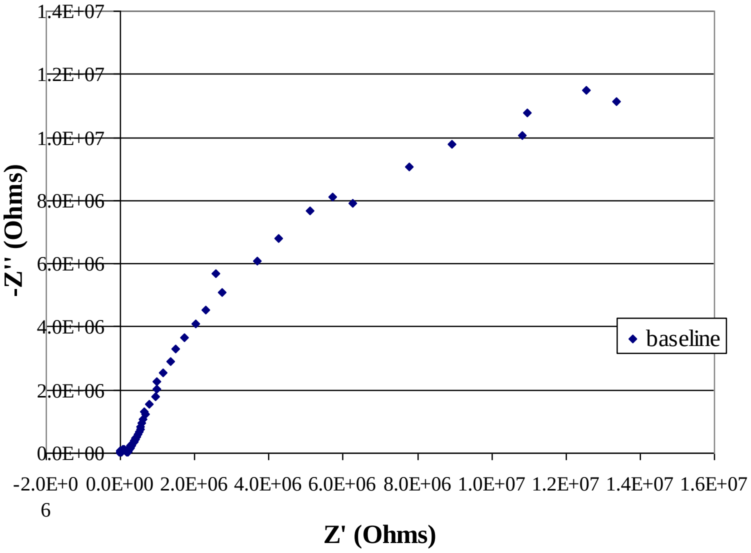

Figure 7: Baseline of cell Nyquist plot before dendritic growth

Figure 8 shows the Nyquist plot during the dendritic growth. It can be seen that the two semicircles in the plot were shrinking on both real and imaginary axis in general as dendrites grew longer and denser, although in the middle of the process this shrinking trend may have reversed a couple of times. The first semicircle of baseline (no dendritic growth) was larger than all the successive first semicircles during dendritic growth, and the second semicircle of baseline (shown more clearly in Figure 7) was also much larger than those second semicircles during dendritic growth. In addition, the dramatic change of the second semicircle on the Nyquist plot (more than one order of magnitude) can be possibly used as a forerunner signal prior to dendritic growth. Table 1 shows the characteristic frequencies (Hz) according to the top point of the first semicircle in Figure 8.

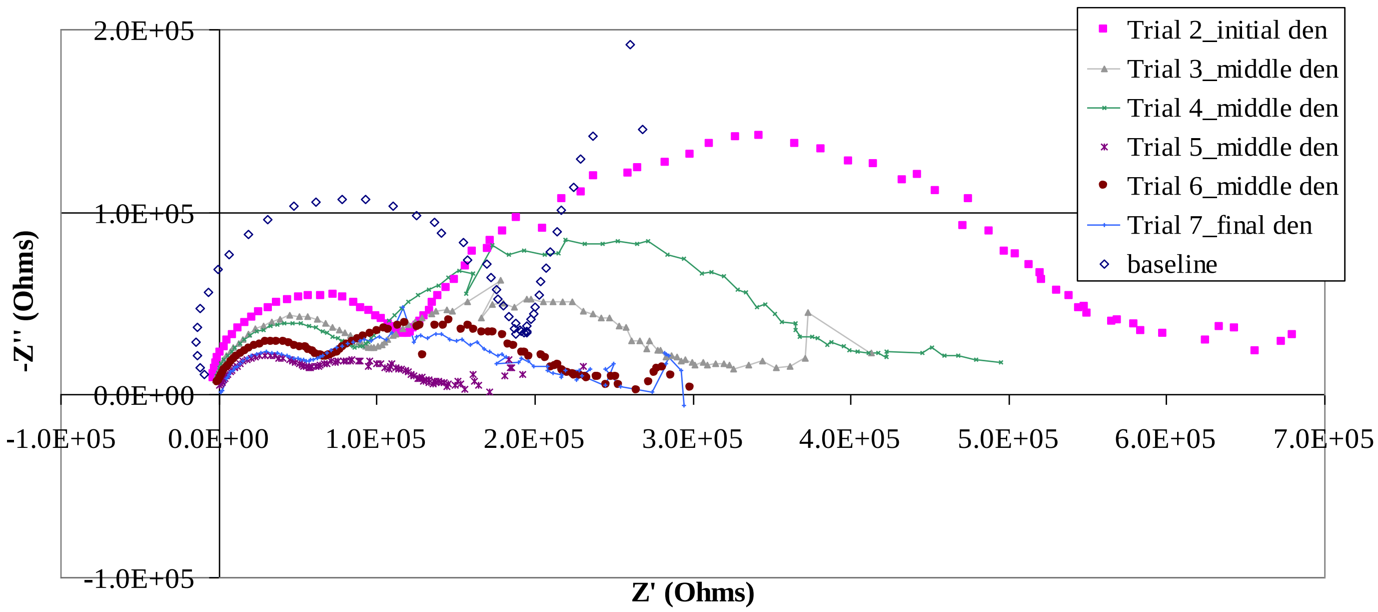

Figure 8: Nyquist plot during dendritic growth

Table 1 Characteristic Frequencies (Hz) according to the Top Point of the First Semicircle in Figure 8

Baseline	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
$1.36x10^4$	$5.41x10^3$	$6.3x10^3$	$7.36x10^3$	$5.41x10^3$	$7.36x10^3$	$5.41x10^3$

Based on Figure 8, the solution resistance R_c can be obtained, which is the real part of the right crossing point between the first semicircle and x axis. Table 2 shows the actual values of solution resistances. It can be seen that the solution resistance was decreasing during the dendritic growth, as shown in Figure 9, since more and more copper ions dissolved into the solution and increased the concentration of migrating ions.

Table 2 Solution Resistance R_c (Ohms) based on Figure 8

Baseline	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
$1.88x10^5$	$1.20x10^5$	$9.43x10^4$	$8.57x10^4$	$5.79x10^4$	$7.1x10^4$	$5.52x10^4$

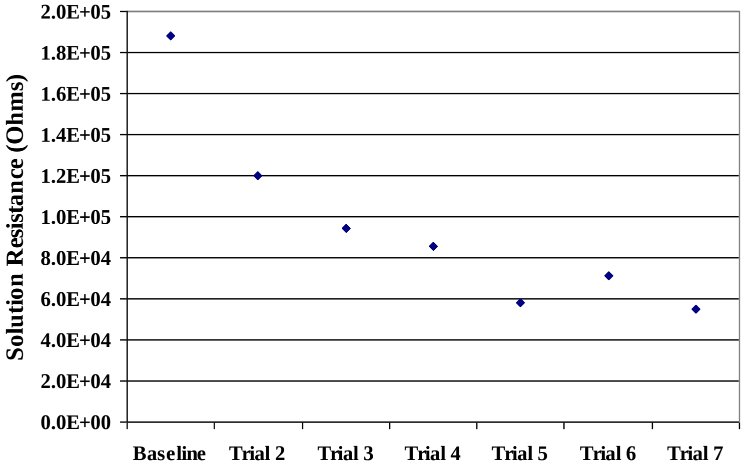

Figure 9: Change of solution resistance R_c during dendritic growth based on Table 2

Charge transfer resistance was also obtained based on Figure 8 and shown in Table 3 and Figure 10. It can be seen that the charge transfer resistance was generally decreasing during the dendritic growth, though it could reverse a little in the middle of the process. The reason was because the active surface area of the anode for metal dissolution was increasing once metal dissolved, creating micro-level scours, pits or cracks on the surface, thus making charge transfer process easier.

Table 3 Charge Transfer Resistance Rct (Ohms) based on Figure 8

Baseline	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
$>1.4x10^7$	$5.36x10^5$	$2.58x10^5$	$3.37x10^5$	$8.71x10^4$	$1.94x10^5$	$2.19x10^5$

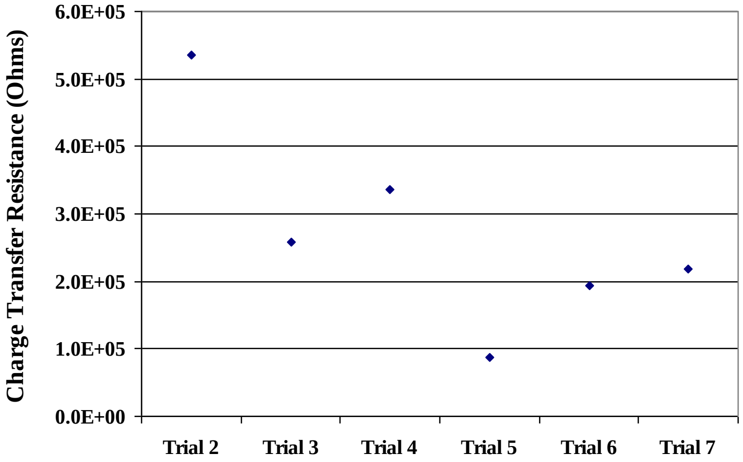

Figure 10: Change of charge transfer resistance Rct during dendritic growth based on Table 3

Based on Equation 1, the double layer charging capacitance is calculated by the charge transfer resistance R_{ct} and f^* (the characteristic frequency for the second semicircle in Figure 8, shown in Table 4). Table 5 and Figure 11 show the actual values and kinetics of C_{dl}.

$$C_{dl} = \frac{1}{2\pi R_{ct} f^*} \tag{1}$$

So the double layer charging capacitance oscillated and did not change much during the dendritic growth.

Table 4 Characteristic Frequencies (Hz) f* according to the Top Point of the Second Semicircle in Figure 8

Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
11.7	63.1	25.1	117	34.1	63.1

Table 5 Change of Double Layer Charging Capacitance Cdl (F) based on Figure 8

Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
2.54×10^{-8}	9.78×10^{-9}	1.88×10^{-8}	1.56×10^{-8}	2.41×10^{-8}	1.15×10^{-8}

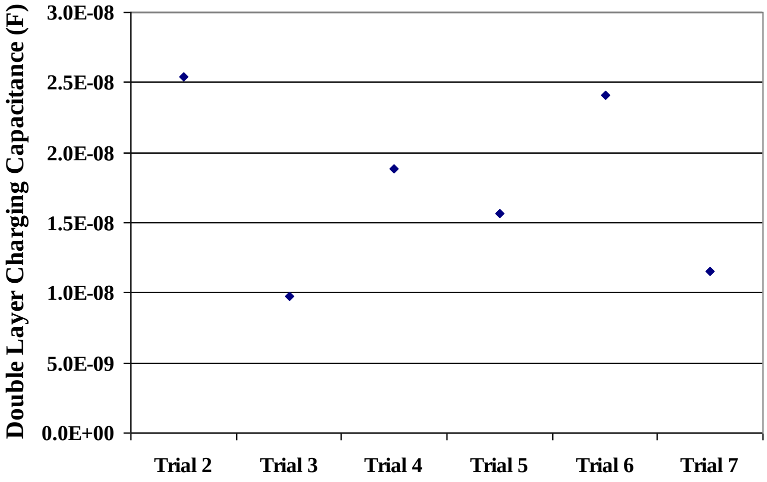

Figure 11: Change of double layer capacitance C_{dl} during dendritic growth based on Table 5

3.2 A Simulation of Impedance Kinetics based on the Equivalent Circuit Model Shown in Figure 5

The paralllel circuit inside the dotted lines in Figure 5 represents the electrochemical process at the electrode-solution interfaces, and its impedance Z_{reac} is shown in Equation 2. So the overall impedance of the cell Z is the overall parallel circuit containing C_c, Z_{reac}, and R_c, as shown in Equation 3.

$$Z_{reac} = R_c + \frac{\frac{1}{j\omega C_{dl}}(R_{ct} + \sigma\omega^{-1/2}(1-j))}{\frac{1}{j\omega C_{dl}} + R_{ct} + \sigma\omega^{-1/2}(1-j)} \tag{2}$$

$$= R_c + \frac{R_{ct} + \sigma\omega^{-1/2}}{(1+\sigma\omega^{1/2}C_{dl})^2 + (R_{ct}\omega C_{dl} + \sigma\omega^{1/2}C_{dl})^2} - \frac{\sigma\omega^{-1/2} + 2\sigma^2 C_{dl} + R_{ct}C_{dl}(\sigma + \sigma\omega^{1/2} + R_{ct}\omega)}{(1+\sigma\omega^{1/2}C_{dl})^2 + (R_{ct}\omega C_{dl} + \sigma\omega^{1/2}C_{dl})^2} j$$

$$Z = \frac{\frac{1}{j\omega C_c} Z_{reac}}{\frac{1}{j\omega C_c} + Z_{reac}} \tag{3}$$

Based on Equation 3 and the extracted cell parameters from Figure 8 such as the C_c, R_c , C_{dl}, and R_{ct}, a simulation was conducted and the result was shown in Figure 12. The simulated result generally confirms the experimental data. It can be seen that the two semicircles generally shrink although they reverse a little in the middle of the process.

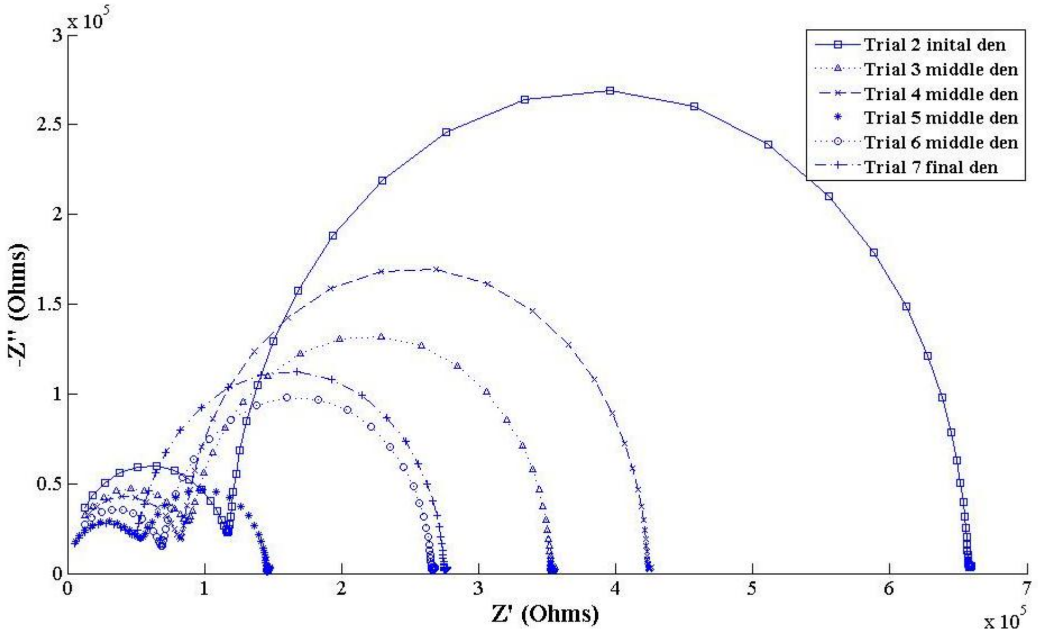

Figure 12: Simulated Nyquist plot during dendritic growth based on the extracted values C_c, R_c , C_{dl}, and R_{ct} from Figure 8

3.3 Frequency Dependance of Impedance Magnitude and Phase Shift

The magnitude of impedance generally shows the characteristic feature of a resistor-capacitor parallel circuit, shown in Figure 13. In the high frequency region, the capacitance dominates, while at low frequency region, the resistance dominates. At low frequency region the impedance approximately equals the sum of R_c and R_{ct}.

Figure 14 shows the phase shift dependency on frequency. Since the cell generally behaves as a resistor-capacitor parallel circuit, the phase shift at low frequency region shows a resistance feature and is close to 0°. While at high frequency region the phase shift shows a capacitance feature and is close to -90°. In the middle frequency range (around 1000 Hz) the peak was caused by the interaction of the two parallel circuits (C_c-R_c and C_{dl}-R_{ct}). Figure 15 shows the final dendrites grown on the boards.

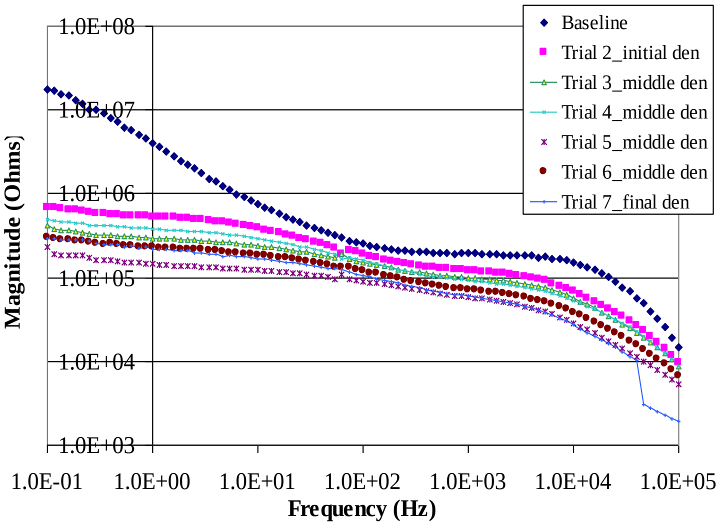

Figure 13: Bode plot during dendritic growth

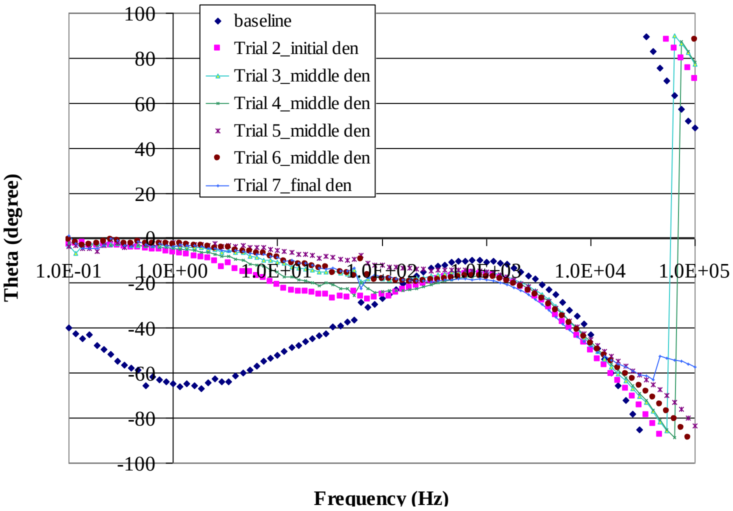

Figure 14: Phase angle plot during dendritic growth

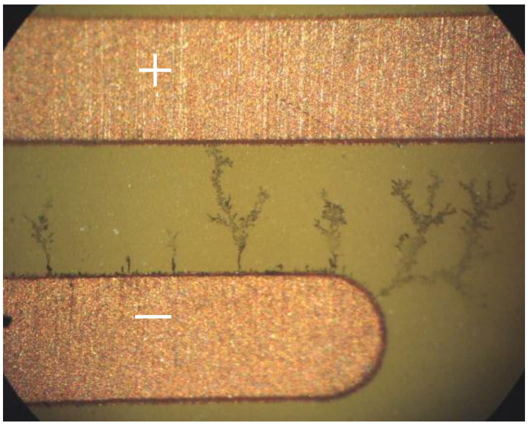

Figure 15: The final dendrites grown after the cyclic DC and AC applications.

4. Conclusions

The kinetics of electrochemical migration in bulk deionized water was investigated by using AC impedance technique. This approach was effective in extracting the cell parameters during the electrochemical process. A physicochemical model containing two time constants (two resistor-capacitor parallel circuits) was used to represent the ECM process, and this model was confirmed by simulation results. Further experimental work can be conducted to confirm the correspondence between the EIS spectra and the cell parameters.

Acknowledgements

This research was supported by the members of the CALCE Electronic Products and Systems Consortium at the University of Maryland, College Park.

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Characterization of Electrochemical Migration on Printed Circuit Boards Using Impedance Measurement

Xiaofei He, Dr. Michael H. Azarian, Dr. Michael Pecht

Objective:

- Differentiate individual steps in the mechanisms of electrochemical migration on PCBs using electrochemical impedance spectroscopy (EIS).
- Identify the kinetics of electrochemical system parameters

Introduction to Electrochemical Migration

Electrochemical Migration (ECM) is the growth of conductive metal dendrites through an electrolyte solution under the influence of a DC voltage bias [1].

at anode:

$$Me \rightarrow Me^{x+} + xe^{-}$$

at cathode:

$$Me^{x+} + xe^{-} \rightarrow Me$$

ECM process: path formation, electro-dissolution, ion transportation, electro-deposition, and filament formation [2].

Problem Statement

- Surface insulation resistance (SIR) measurement is a traditional means to determine the susceptibility of printed circuit boards (PCBs) to ECM and the degree of metal migration prior to the failure of PCBs, but it cannot differentiate the individual electrochemical constituents contributing to ECM and identify the dominant ones at different dendritic growth stages.
- Identification of the rate limiting step in the ECM is essential for developing an accurate model of the time-to-failure, and helpful for devising strategies to eliminate or mitigate ECM.
- Despite its significance, there have been few published studies that analyze the kinetics of this failure mechanism in terms of its constituent steps.

Introduction to Electrochemical Impedance Spectroscopy

- Electrochemical impedance spectroscopy (EIS) involves sweeping an AC signal over a wide range of frequencies and measuring the responses of an electrochemical system, thus providing information on the constituent electrochemical processes.
- From the applied perturbation (voltage) and the measured response (current) the magnitude of the impedance and phase shift as a function of the frequency can be determined.
- Results of EIS measurement: impedance represented by Nyquist plot and Bode plot. Analysis of the results allows creation of an equivalent circuit model to describe the electrochemical system.

Source: [3][4][5][6]

EIS Data Representation

- Nyquist Plot

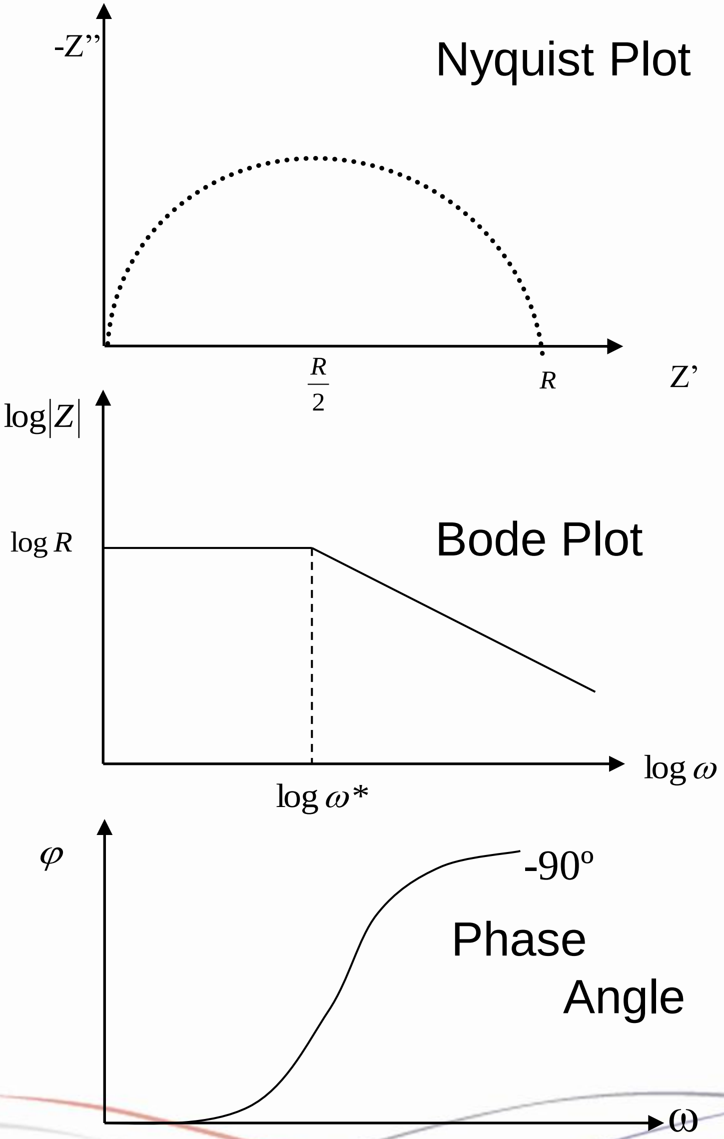
  The impedance contains a real part and an imaginary part:

  Z(ω)=Z' + jZ''=Z'(ω)+ jZ''(ω)

  where *ω* is the frequency. If the real part is plotted on the *real* axis and the conjugate imaginary part on the *imaginary* axis of a chart, a "Nyquist plot“ is obtained.
- Bode Plot

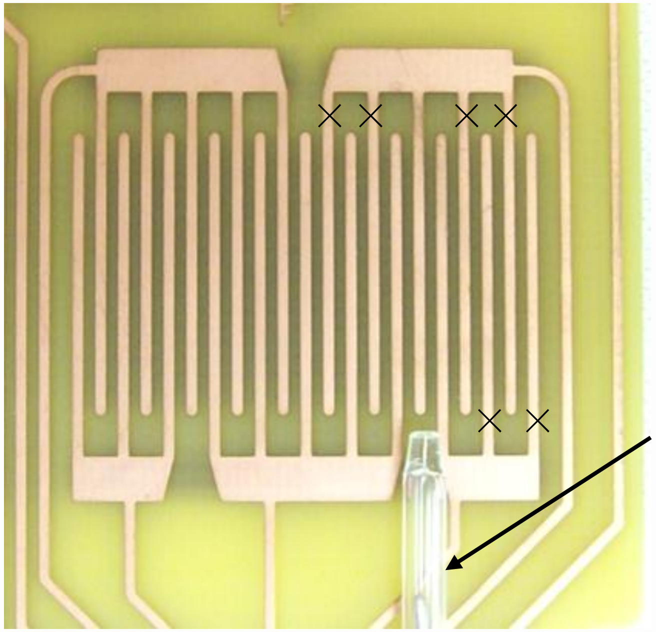
  The chart with the magnitude of *Z* versus the frequencies of perturbations.
- Phase angle

  $$\varphi = \arctan(Z''/Z')$$



Source: [3][4][5][6]

Electrochemical Migration Test Configuration for EIS



Ag/AgCl Micro-Reference Electrode

Test Comb Structure

Tip of Micro-Reference Electrode

Anode

Cathode

Electrodissolution

Ion Transportation

Electrodeposition

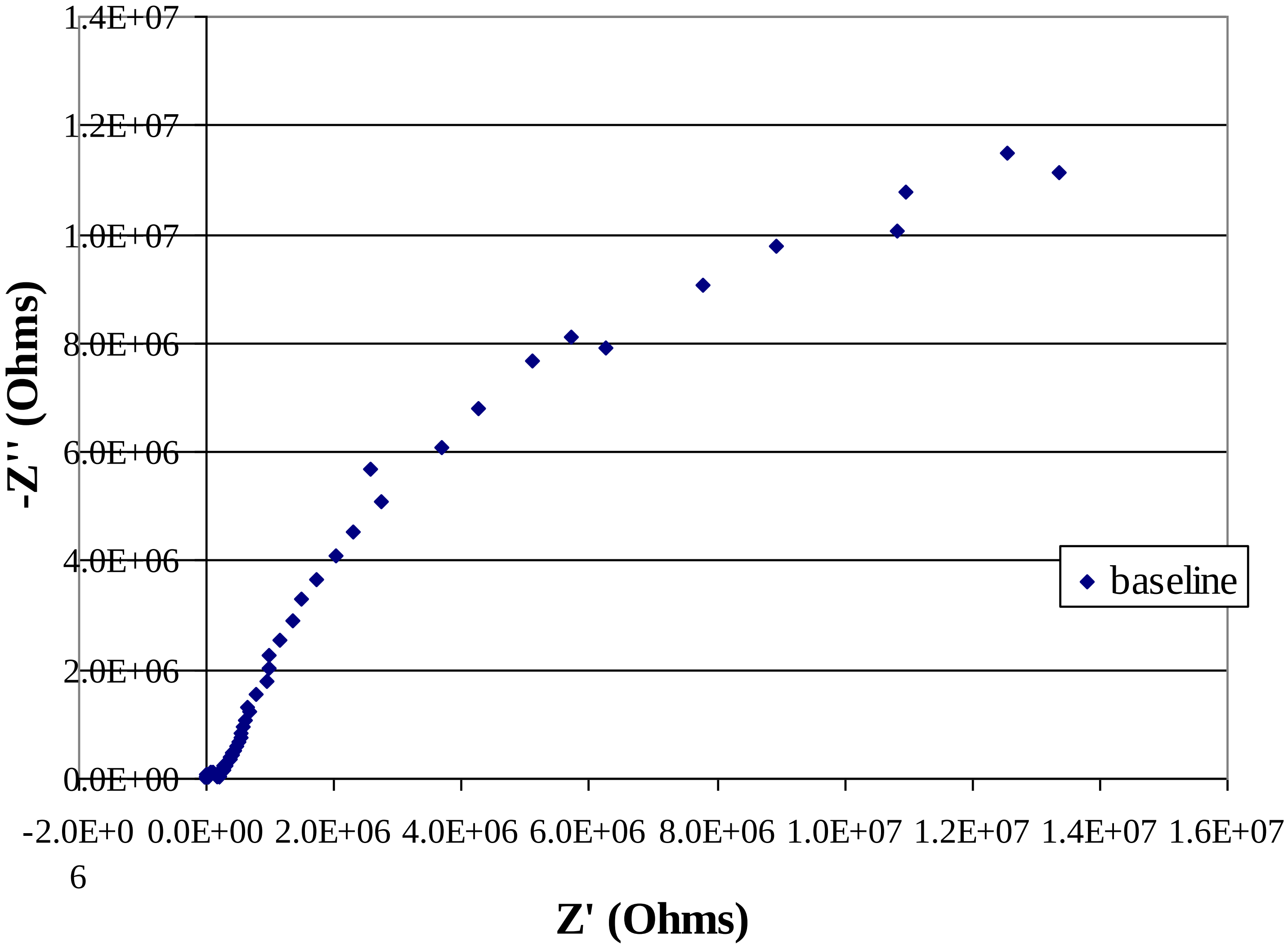
Experimental Conditions

- DI water was used as the electrolyte, and was exposed to the room ambient. Solartron Analytical potentiostat 1287A was used to measure impedance.
- The comb structure was rinsed in DI water for 5 minutes before being inserted into the electrolyte.
- An Ag/AgCl reference electrode (0.21V vs. standard hydrogen electrode) was used to monitor the system potential profile.
- The open circuit potential (OCP), which is the equilibrium potential of the whole system when the external current is zero, of bare copper electrodes, was measured to be 0.05 V~0.08 V relative to the Ag/AgCl reference electrode.

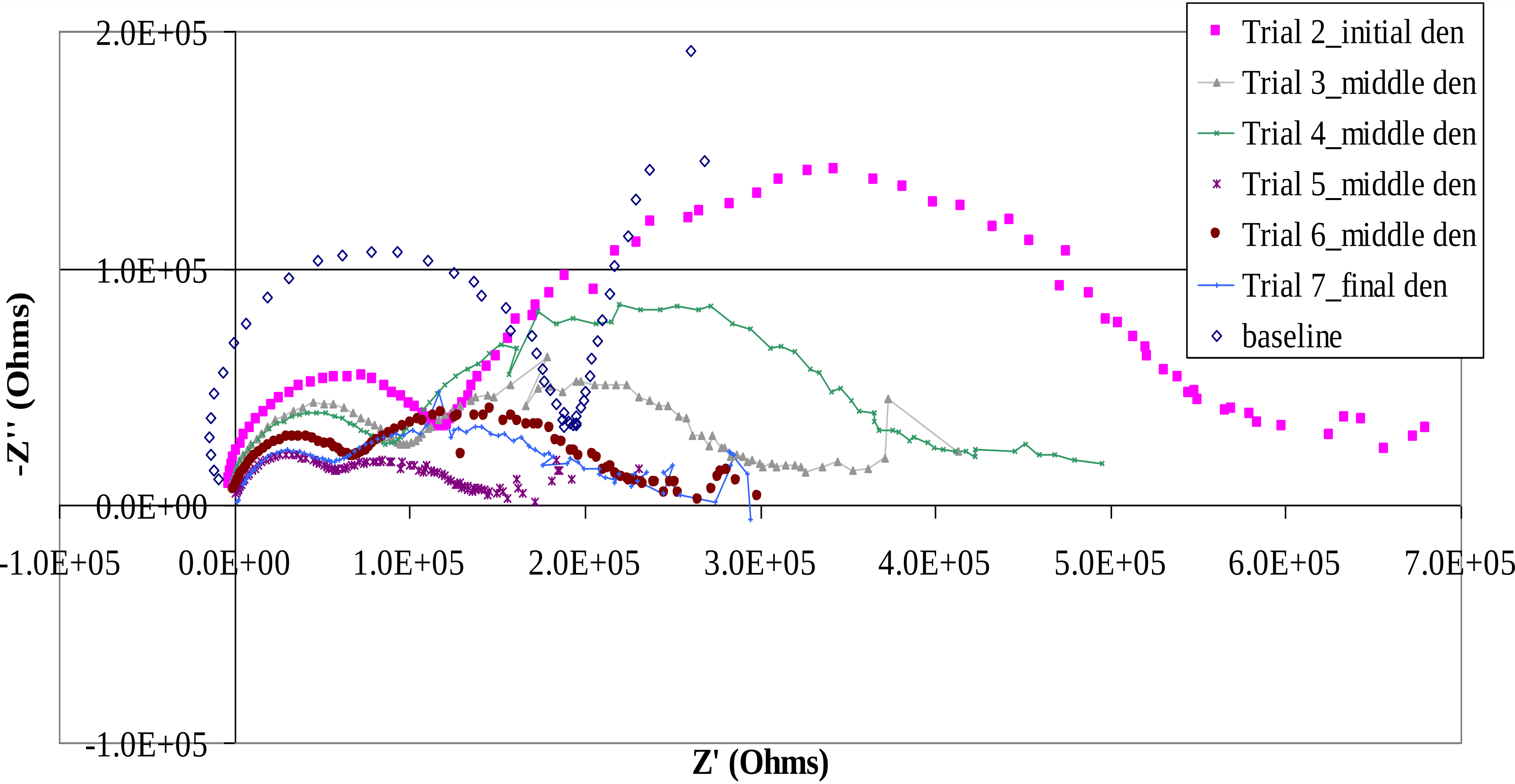
Experimental Procedure

- Rinse the comb structure, put it into the DI water, and locate the Ag/AgCl reference electrode properly.
- Apply a 5 mV AC signal and measure impedance and record the system baseline, based on the OCP. The frequency ranges from 0.1 Hz to 100,000 Hz.
- Apply 1.5 V~2.2 V DC voltage across the anode and the reference electrode, allow dendrites to initiate and grow.
- Apply a 5 mV AC signal relative to OCP and measure impedance.
- Apply DC and AC signal in a cyclic fashion, to drive dendritic growth and to measure impedance respectively, until the dendrites touch the anode.

Initial Nyquist Plot without Dendritic Growth



Nyquist Plot during Dendritic Growth



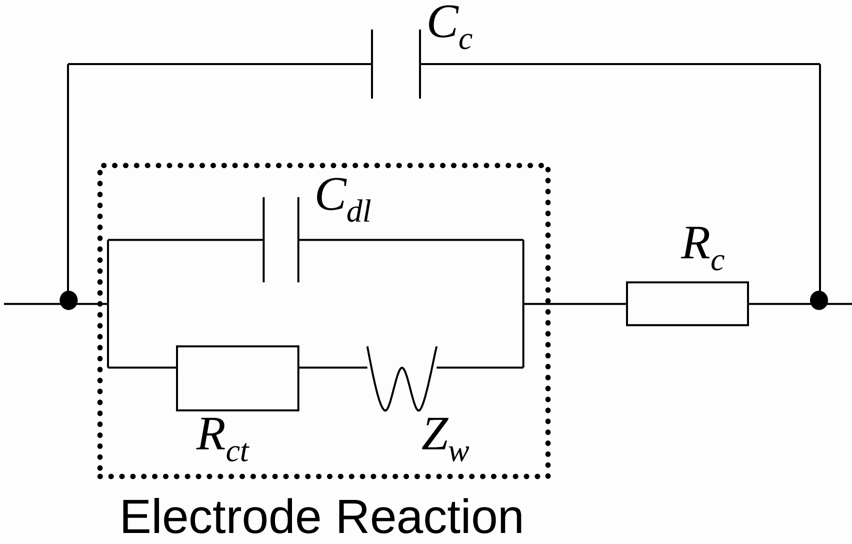
The two semicircles in the plot were shrinking on both real and imaginary axis in general as dendrites grew longer and denser, although in the middle of the process this shrinking trend may have reversed a couple of times.

Equivalent Circuit Components

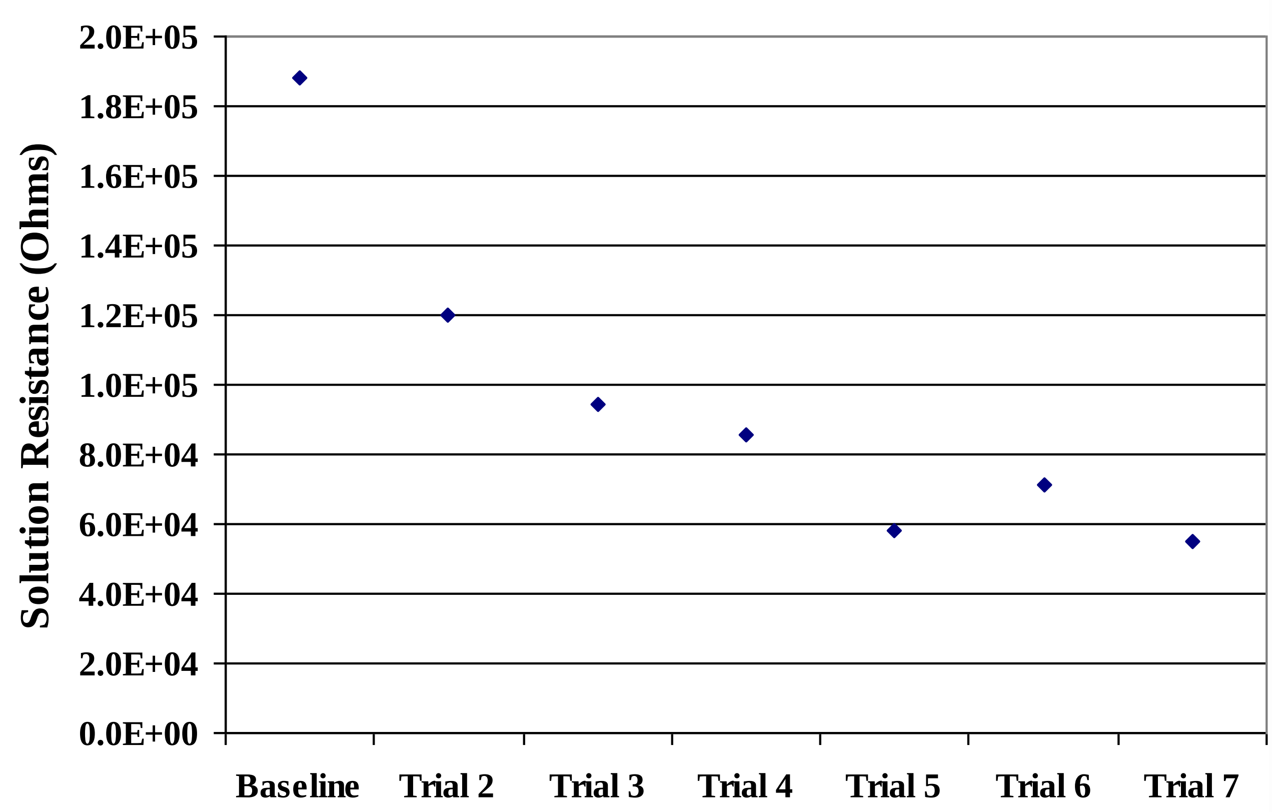
- R_c is the solution resistance. The passage of a current I can generate a voltage drop IR_c.
- R_{ct} is the charge transfer resistance. It is given by

$$R_{ct} = Z_{ct} = \frac{RT}{j_0 zF}$$

  where j_o is exchange current density, which is the current density of an electrochemical system at equilibrium, R is the gas constant, T is the absolute temperature, z is the charge number of the electroactive ions, and F is Faraday constant.
- A circuit dominated by R_{ct} is considered under activation control.
- "Z_W" is Warburg impedance caused by diffusion. A circuit dominated by Z_w is considered under diffusion control.
- C_c is capacitance of comb structures, C_{dl} is double layer capacitance at electrodes.



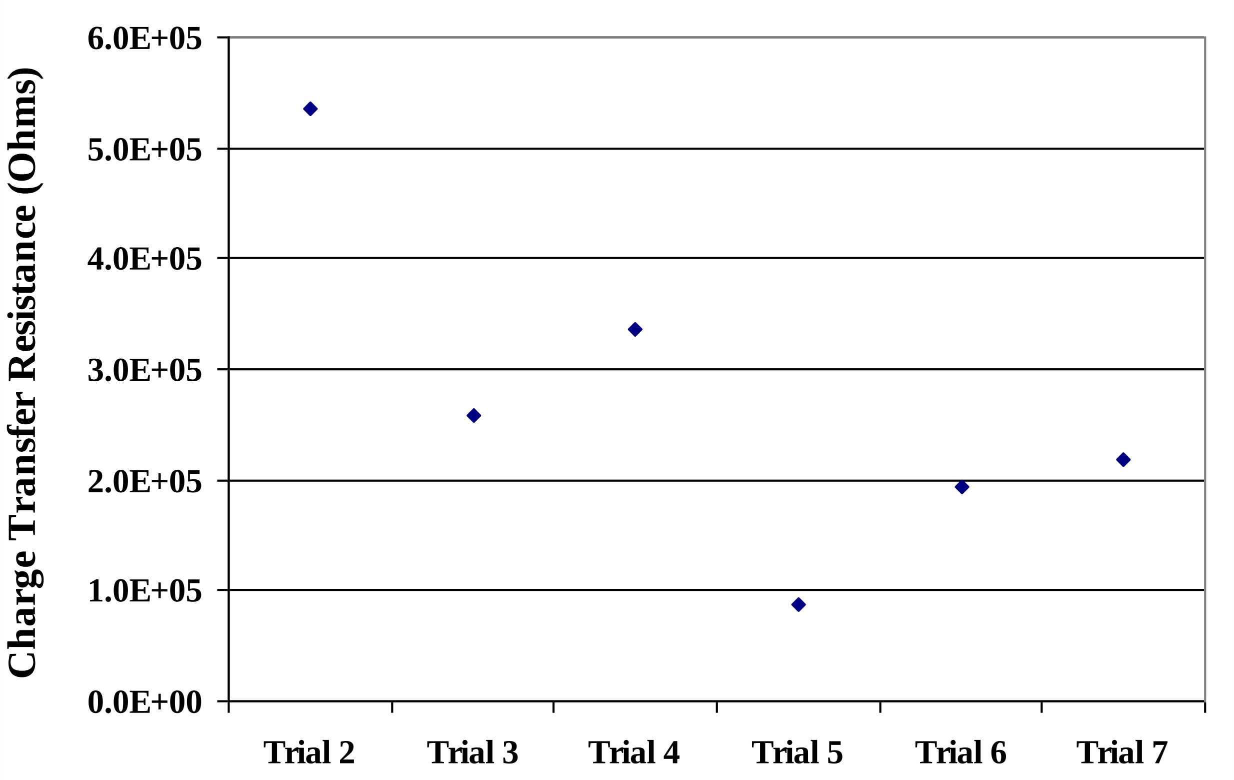
Solution Resistance R_c during Dendritic Growth



Baseline	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
1.88×10^5	1.20×10^5	9.43×10^4	8.57×10^4	5.79×10^4	7.1×10^4	5.52×10^4

Note: in Ohms

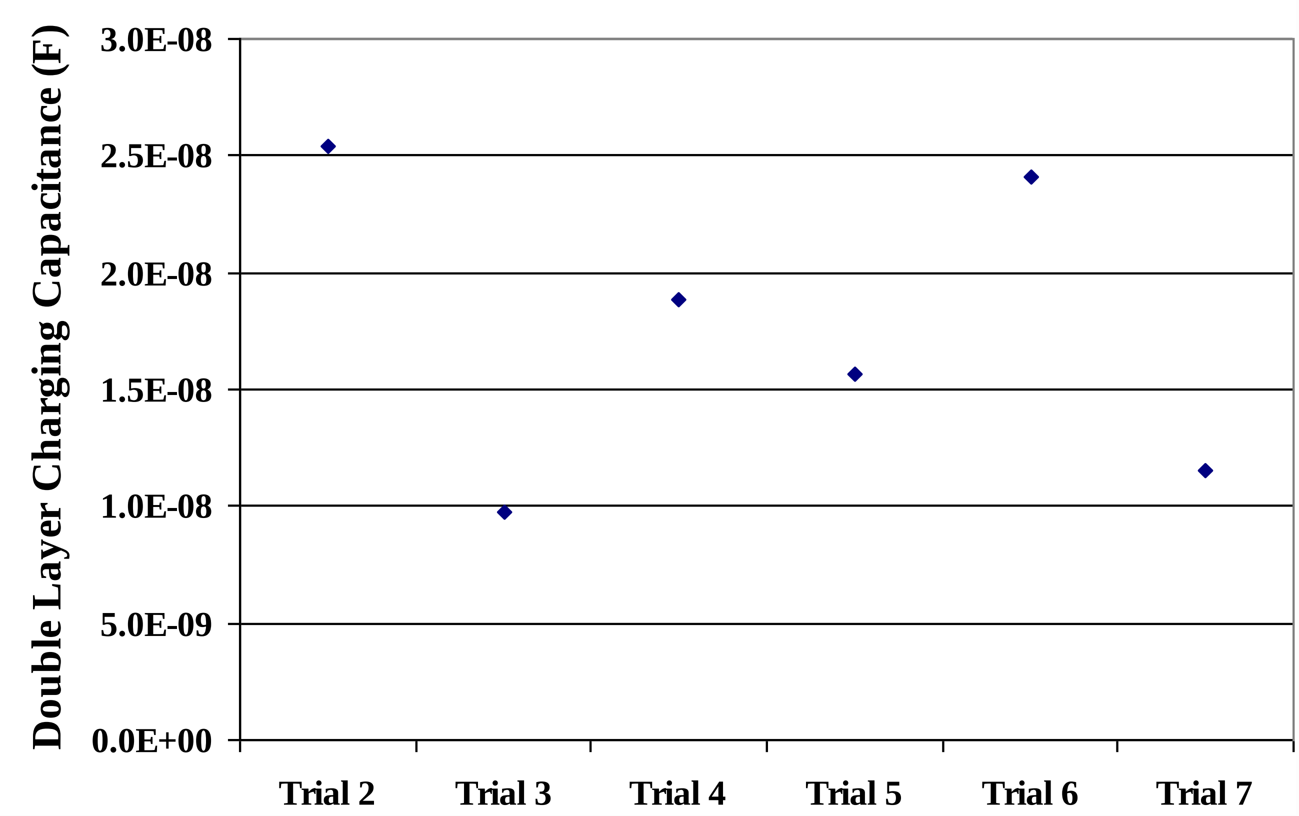
Charge Transfer Resistance R_{ct} during Dendritic Growth



Baseline	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
$>1.4\times10^7$	5.36×10^5	2.58×10^5	3.37×10^5	8.71×10^4	1.94×10^5	2.19×10^5

Note: in Ohms

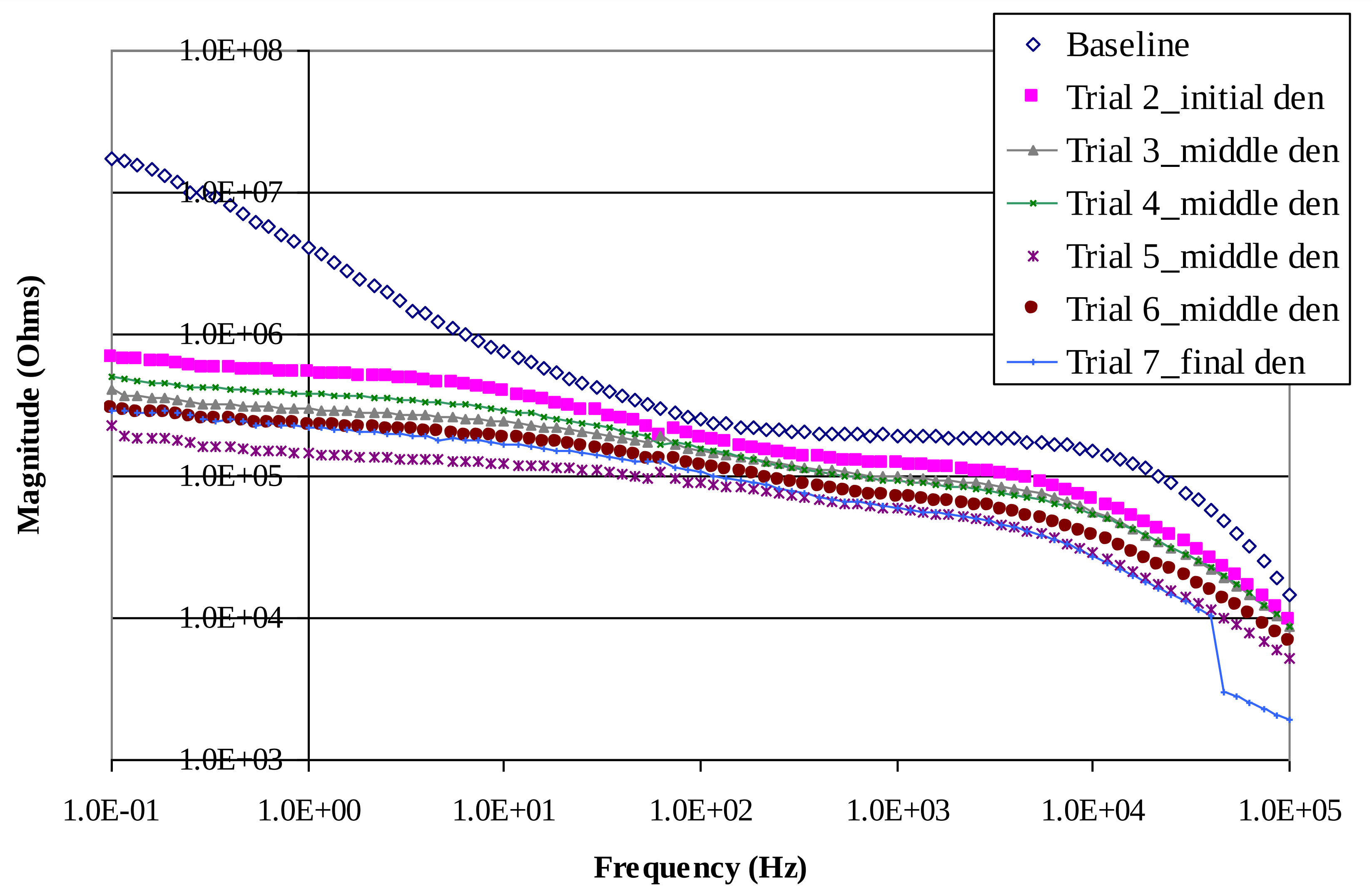
Double Layer Charging Capacitance C_{dl} during Dendritic Growth



Baseline	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7
	2.54×10^{-8}	9.78×10^{-9}	1.88×10^{-8}	1.56×10^{-8}	2.41×10^{-8}	1.15×10^{-8}

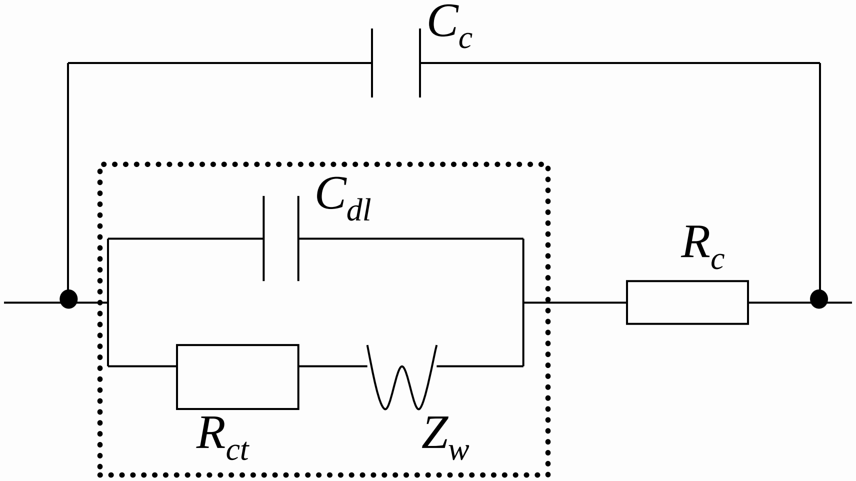
Note: in *F*

Bode Plot during Dendritic Growth



Representation of Electrochemical Cell by An Equivalent Circuit

- The impedance of electrode reaction:



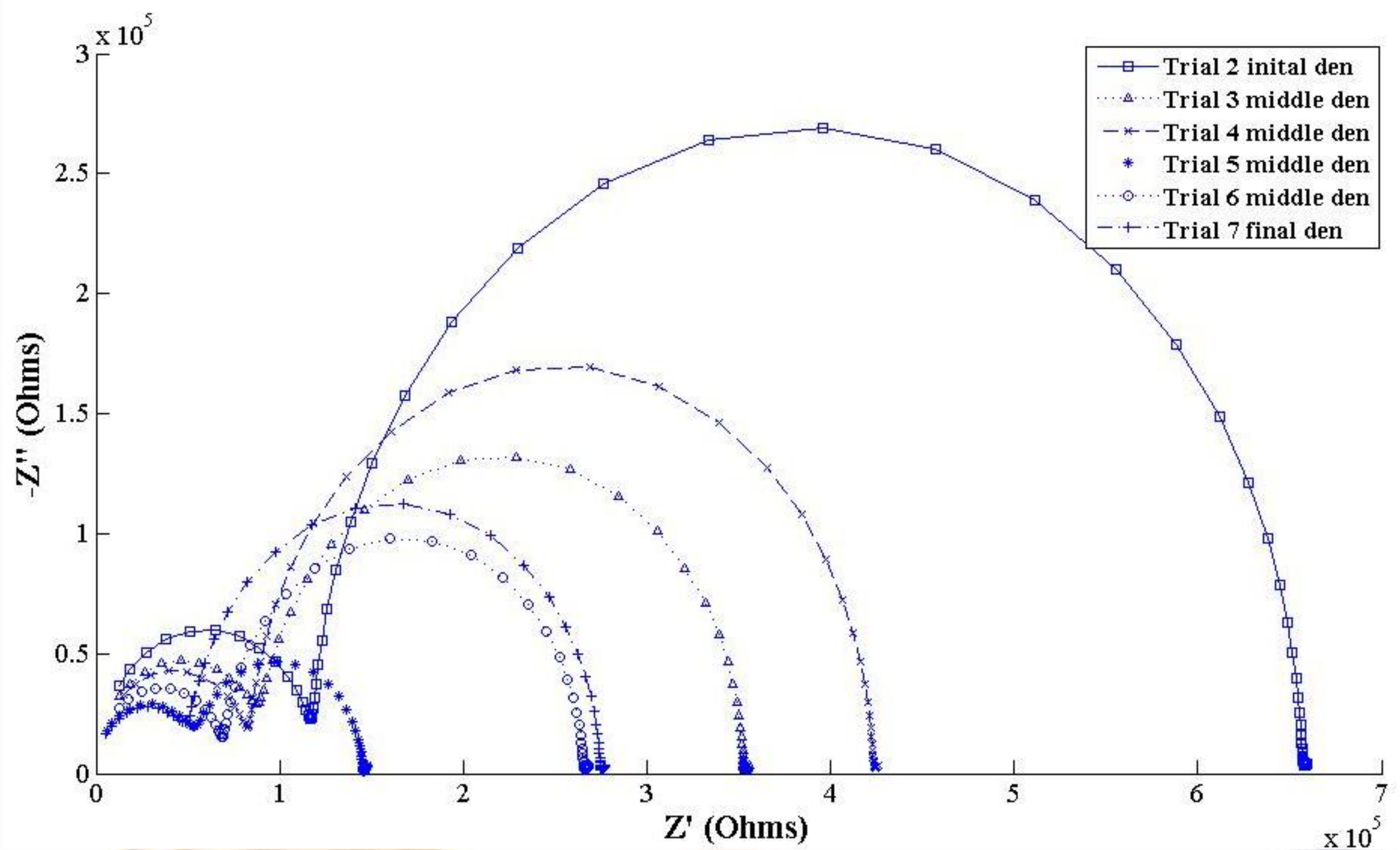
$$Z_{reac} = R_c + \frac{\frac{1}{j\omega C_{dl}}(R_{ct} + \sigma\omega^{-1/2}(1-j))}{\frac{1}{j\omega C_{dl}} + R_{ct} + \sigma\omega^{-1/2}(1-j)}$$

$$= R_c + \frac{R_{ct} + \sigma\omega^{-1/2}}{(1+\sigma\omega^{1/2}C_{dl})^2 + (R_{ct}\omega C_{dl} + \sigma\omega^{1/2}C_{dl})^2} - \frac{\sigma\omega^{-1/2} + 2\sigma^2 C_{dl} + R_{ct}C_{dl}(\sigma + \sigma\omega^{1/2} + R_{ct}\omega)}{(1+\sigma\omega^{1/2}C_{dl})^2 + (R_{ct}\omega C_{dl} + \sigma\omega^{1/2}C_{dl})^2} j$$

- The overall impedance:

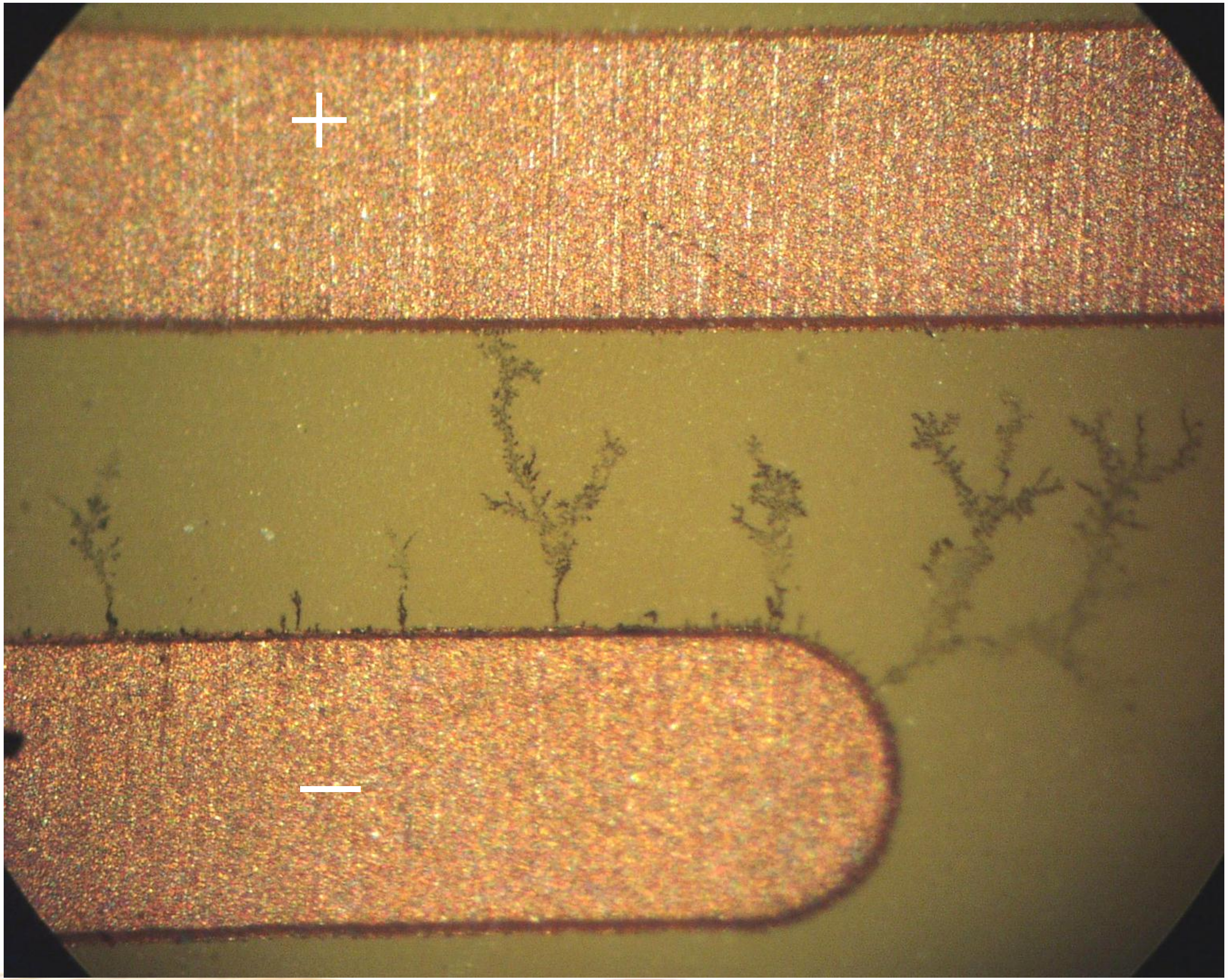
$$Z = \frac{\frac{1}{j\omega C_c} Z_{reac}}{\frac{1}{j\omega C_c} + Z_{reac}}$$

Simulation Based on Extracted Cell Parameters



The two semicircles generally shrink although they reverse a little in the middle of the process, which confirms the experimental data.

The Final Dendrites



Conclusions

- Assuming that solution resistance R_c and capacitance of comb structures C_c dominate the impedance in the high frequency range (>10^3 Hz), the kinetics of the cell parameters during dendritic growth can be obtained:
  - R_c and R_{ct} generally decreased;
  - C_{dl} oscillated and did not change much.
- AC impedance was effective in extracting the electrochemical cell parameters during the electrochemical process.
- A physicochemical model containing two time constants (two resistor-capacitor parallel circuits) was used to represent the ECM process, and this model was confirmed by simulation results.

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