

Fuel Cell Characterization

What Do We Want To Know?

- Overall performance (i-V curve, power density)
- Kinetic properties (η_{act} , j_0 , α , electrochemically active surface area)
- Ohmic properties (R_{ohmic} , electrolyte conductivity, contact resistances, electrode resistances, interconnect resistances)
- Mass transport properties (j_L , D_{eff} , pressure losses, reactant/product homogeneity)
- Parasitic losses (j_{leak} , side reactions, fuel crossover)
- Electrode structure (porosity, tortuosity, conductivity)
- Catalyst structure (thickness, porosity, catalyst loading, particle size, electrochemically active surface area, catalyst utilization, triple phase boundaries, ionic conductivity, electrical conductivity)
- Flow structure (pressure drop, gas distribution, conductivity)
- Heat generation/heat balance
- Lifetime issues (lifetime testing, degradation, cycling, startup/shutdown, failure, corrosion, fatigue)

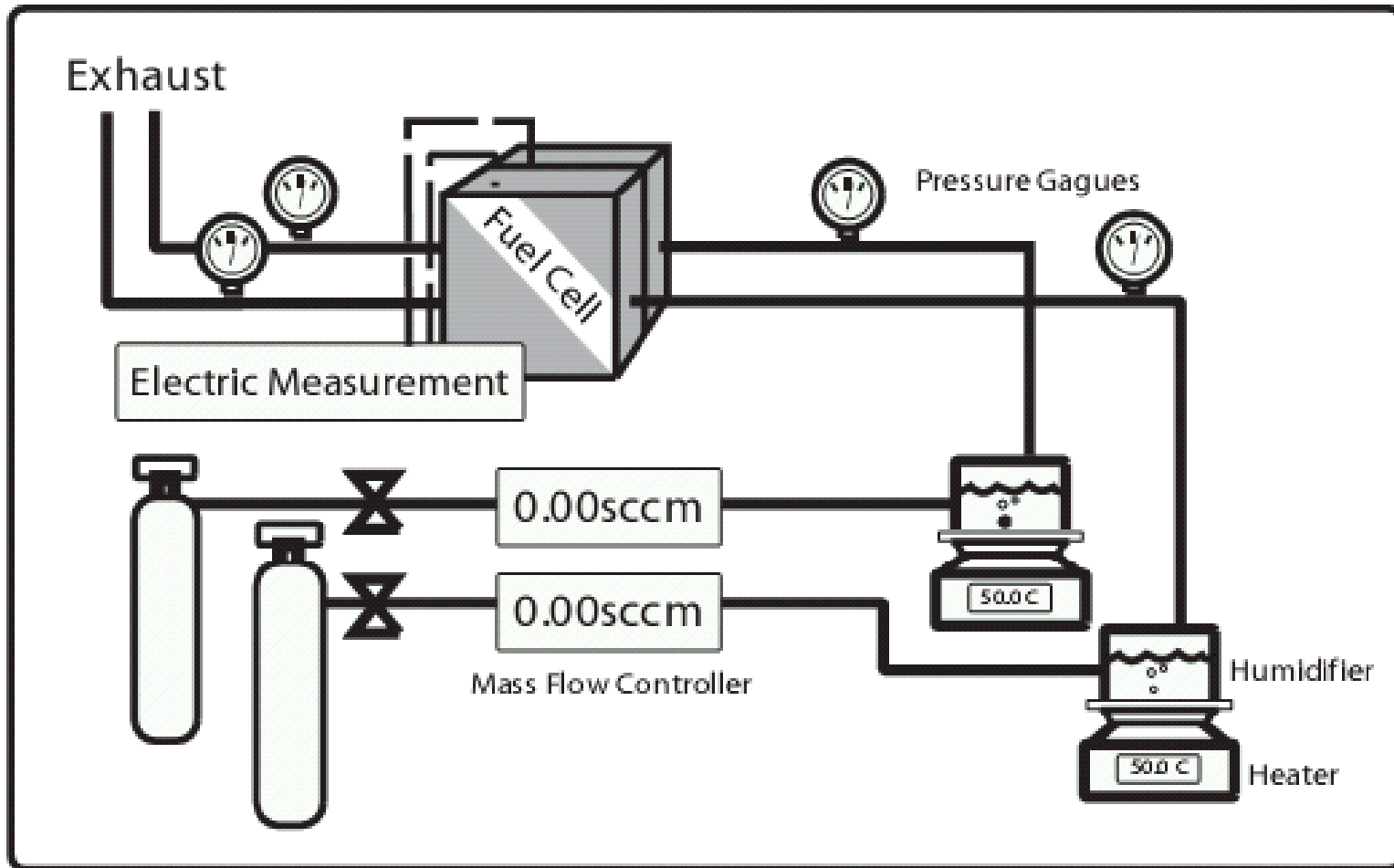
In situ Characterization

1. **Current Voltage (j-V) Measurement:** The most ubiquitous fuel cell characterization technique. A j-V measurement provides an overall quantitative evaluation of fuel cell performance and fuel cell power density.
2. **Current Interrupt Measurement:** Separates the contributions to fuel cell performance from ohmic and non-ohmic processes. Versatile, straightforward, and fast, current interrupt can be used even for high power fuel cell systems and is easily implemented in parallel with j-V curve measurements.
3. **Electrochemical Impedance Spectroscopy (EIS):** A more sophisticated technique that can distinguish between ohmic, activation, and concentration losses. However, the results may be difficult to interpret. EIS is relatively time consuming and difficult to implement for high power fuel cell systems.
4. **Cyclic Voltammetry (CV):** Another sophisticated technique that provides insight into fuel cell reaction kinetics. Like EIS, CV can be time consuming and results may be difficult to interpret. CV may require specialized modification of the fuel cell under test and/or use of additional test gases such as Argon or Nitrogen.

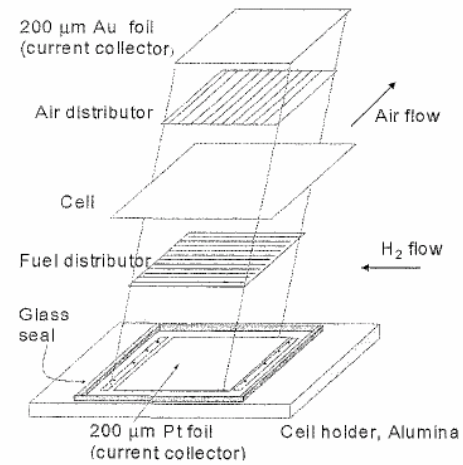
Ex situ Characterization

1. **Porosity Determination:** Effective fuel cell electrode and catalyst structures must have high porosity. Several characterization techniques determine the porosity of sample structures although many of them are destructive tests. More sophisticated techniques even produce approximate pore size distributions.
2. **B.E.T. Surface Area Measurement:** Fuel cell performance critically depends on the use of extremely high surface area catalysts. Some electrochemical techniques yield approximate surface area values, however the B.E.T. method allows highly accurate ex-situ surface area determinations for virtually any type of sample.
3. **Gas Permeability:** Even highly porous fuel cell electrodes may not be very gas-permeable if the pores don't lead anywhere. Understanding mass transport in fuel cell electrodes, therefore, requires permeability measurements in addition to porosity determination. While fuel cell electrodes and catalyst layers should be highly permeable, electrolytes should be gas tight. Gas permeability testing of electrolytes is critical to the development of ultra-thin membranes, where gas leaks can prove catastrophic.
4. **Structure Determinations:** A wide variety of microscopy and diffraction techniques are used to investigate the structure of fuel cell materials. By structure, we mean grain size, crystal structure, orientation, morphology, etc. This determination is especially critical when new catalysts, electrodes, or electrolytes are being developed, or when new processing methods are used.
5. **Chemical Determinations:** In addition to characterizing physical structure, characterizing the chemical composition of fuel cell materials is also critical. Fortunately, many techniques are available for chemical composition and analysis. Often, the hardest part is deciding which is best for a given situation.

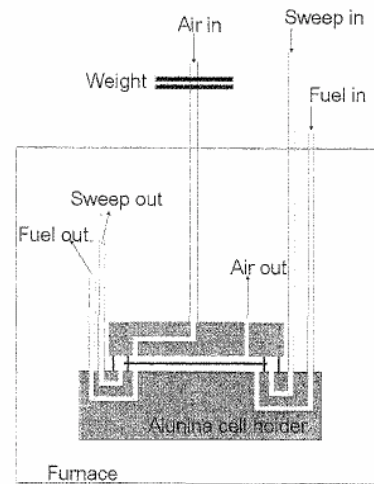
Test Setup



SOFC Test Setup

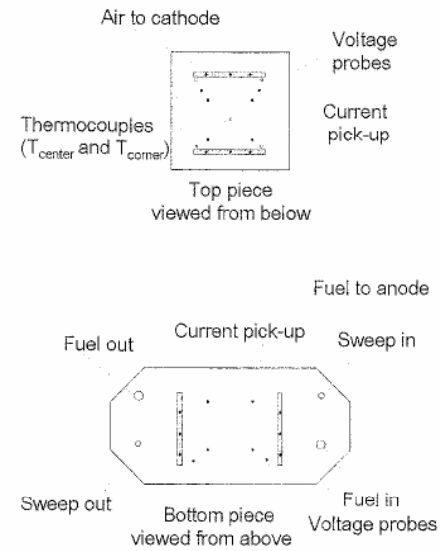


a)



b)

Alumina cell holder

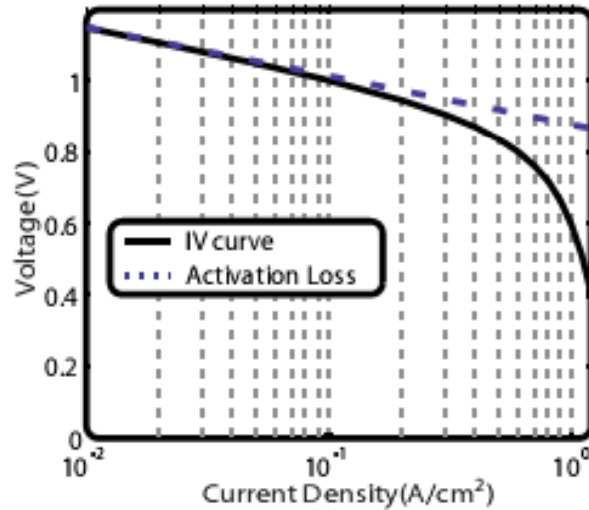


c)

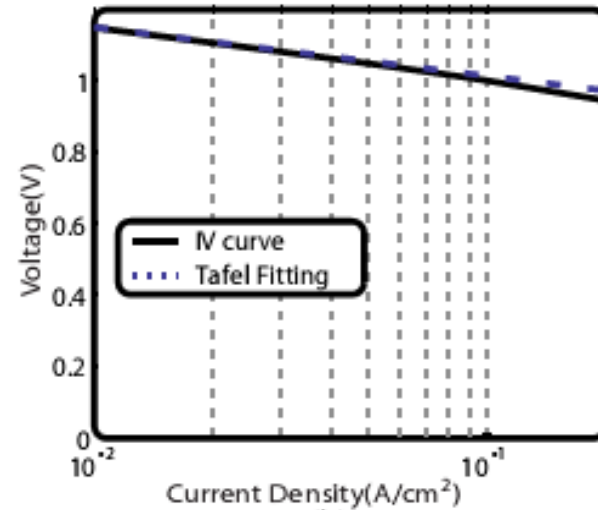
Current Voltage Measurement

- Galvanostat/Potentiostat system
- Steady state
- Test condition
 - Warm-up
 - Temperature
 - Flow rate (stoichiometric number)
 - Compression force

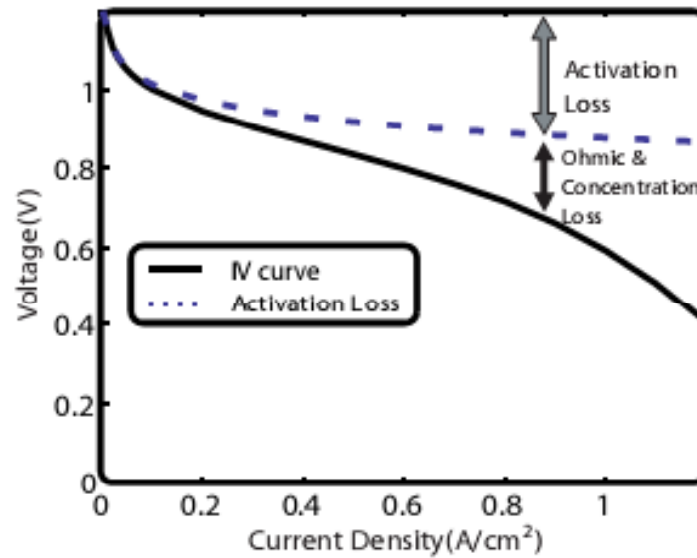
Polarization Curve



(a)

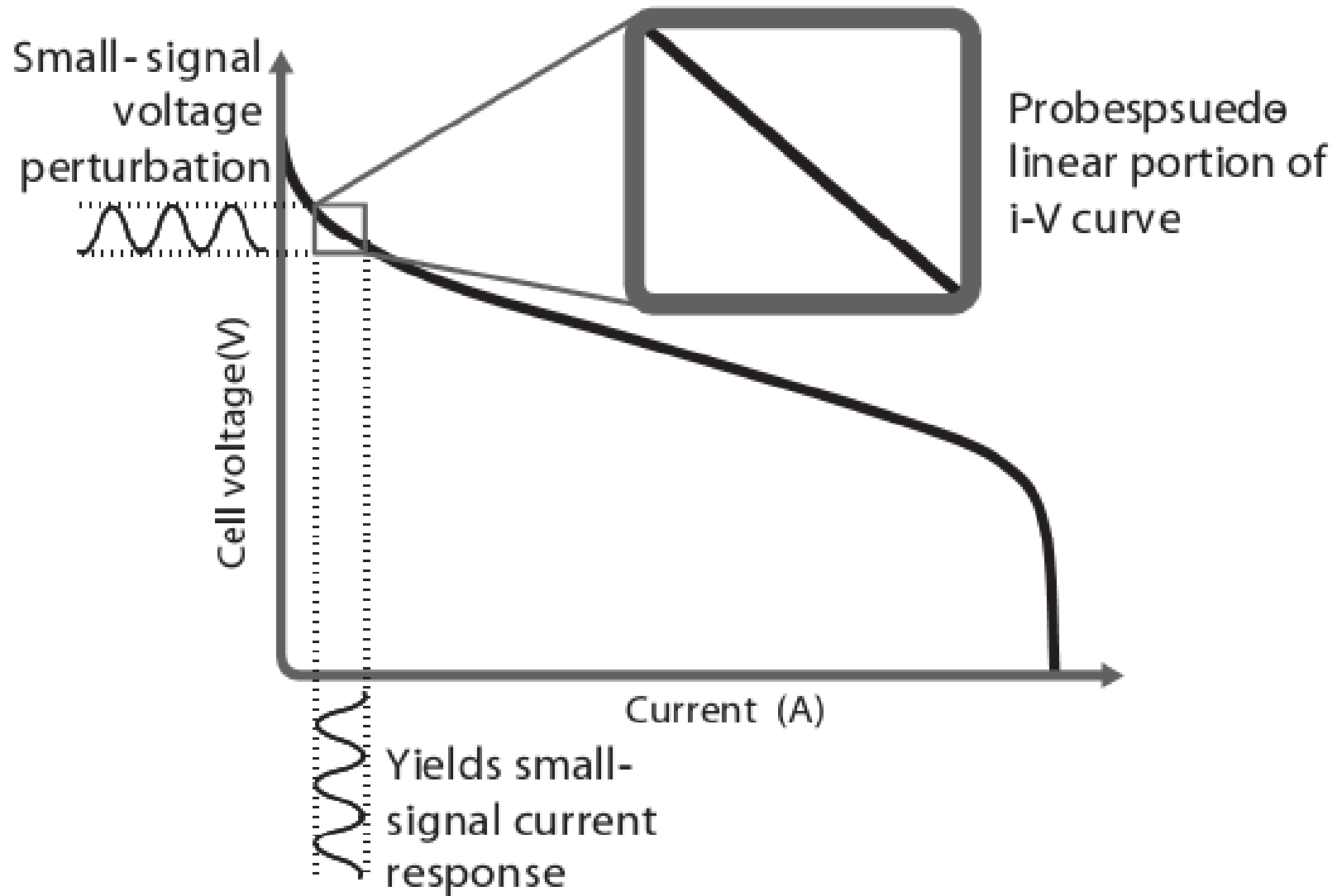


(b)

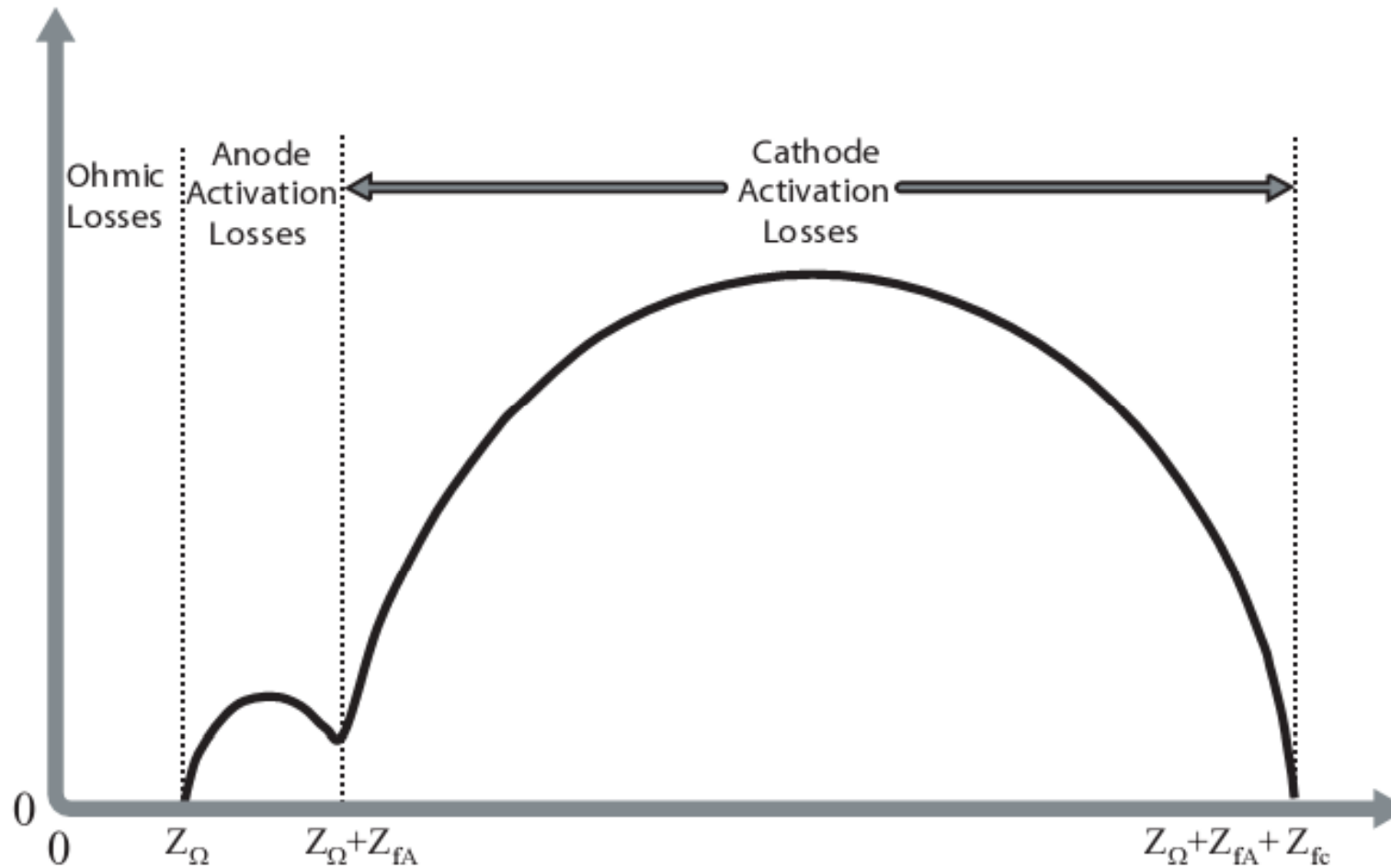


(c)

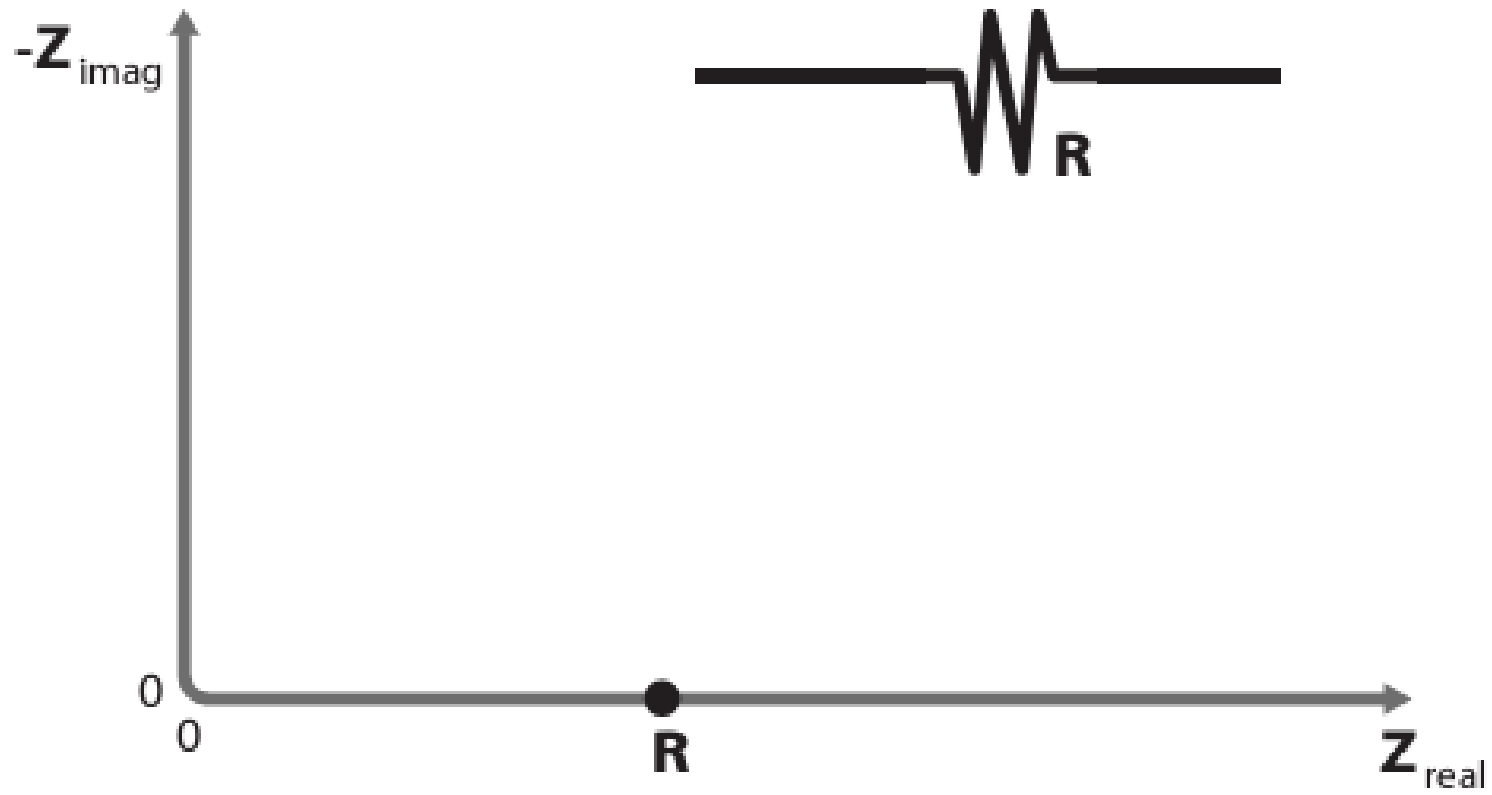
Electrochemical Impedance Spectroscopy



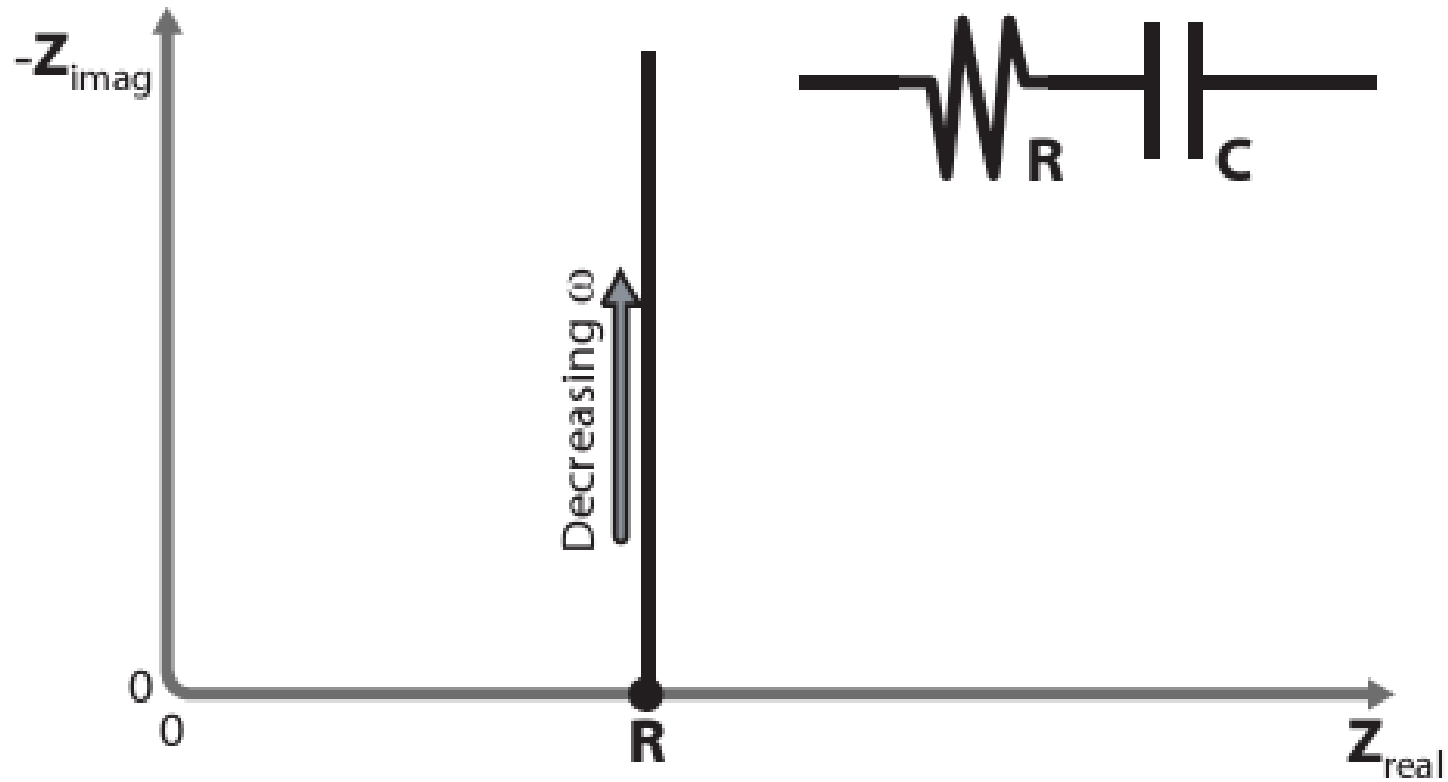
Electrochemical Impedance Spectroscopy



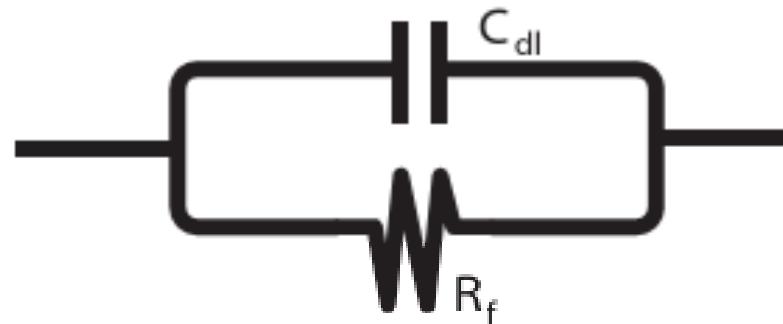
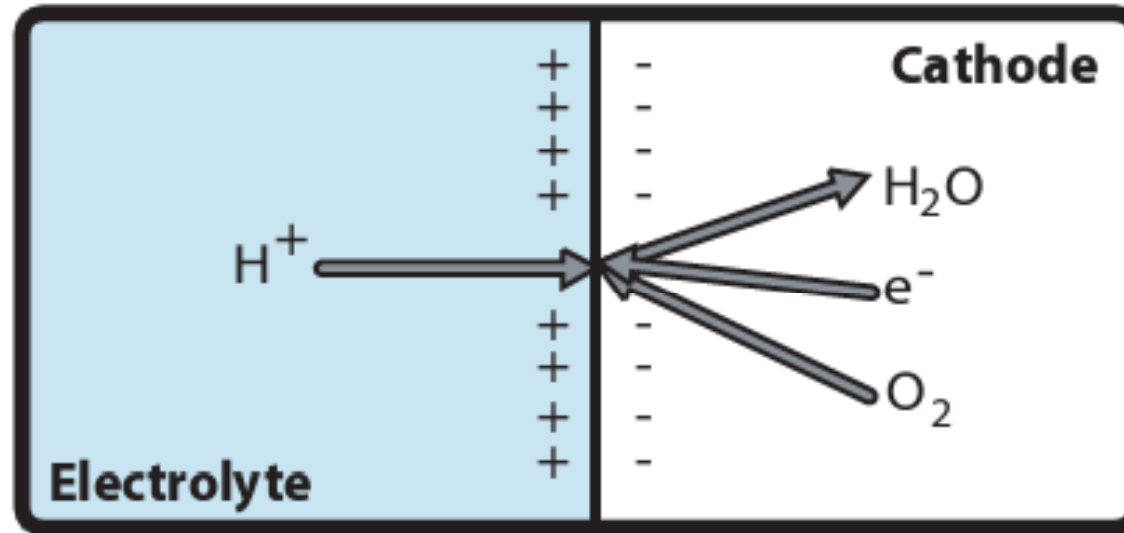
Resistor



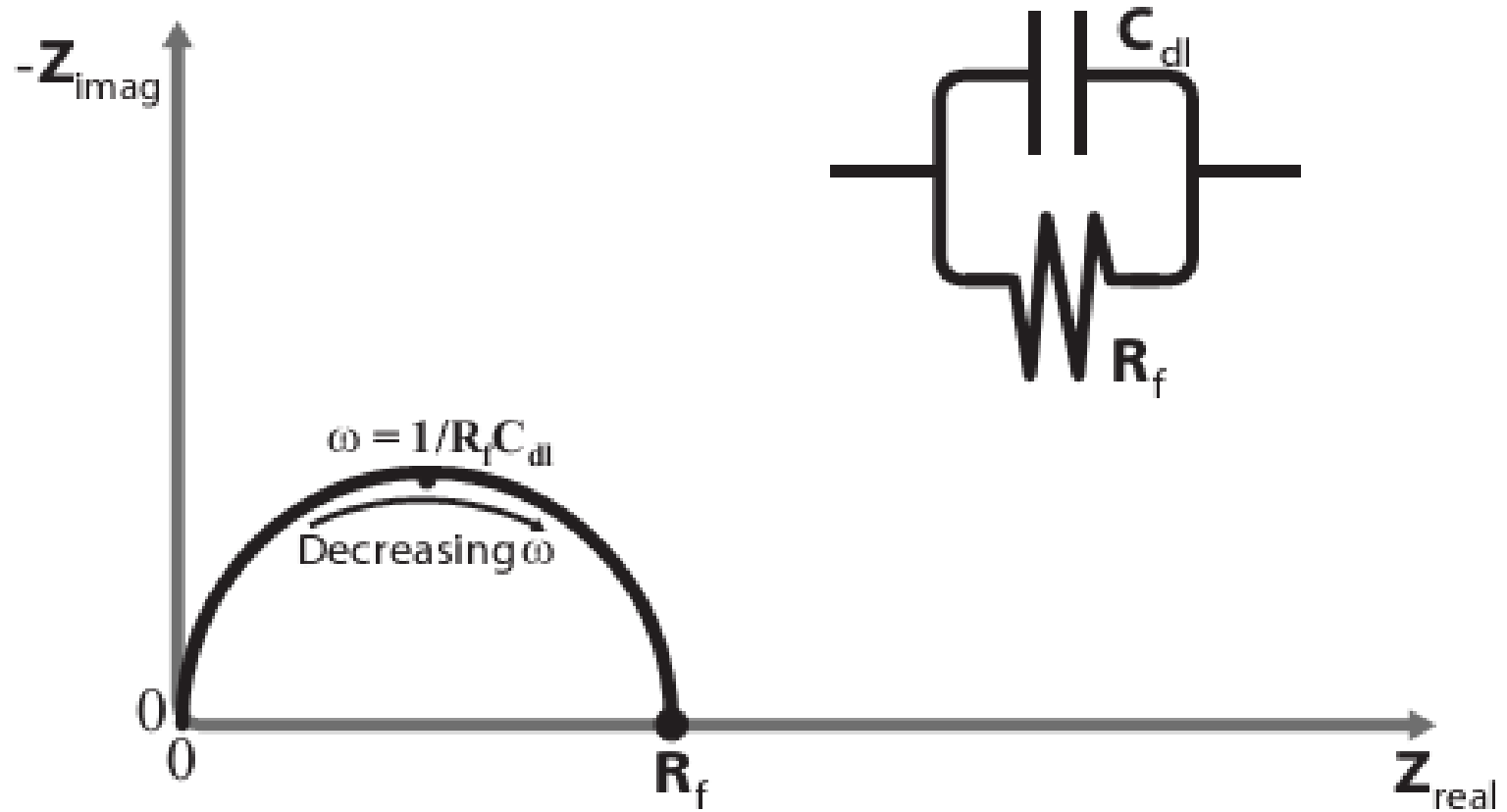
Resistor/Capacitor



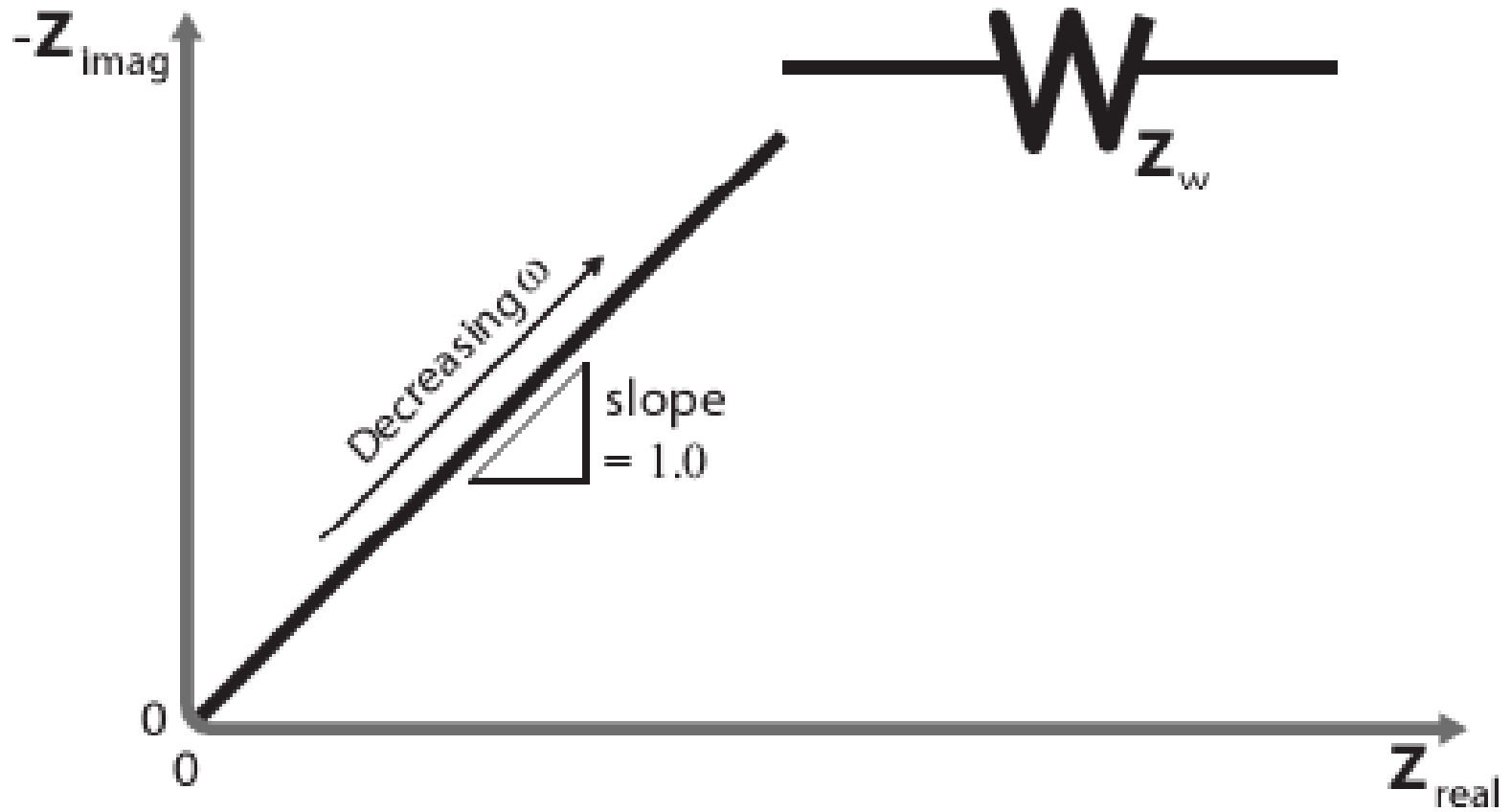
Resistor/Capacitor



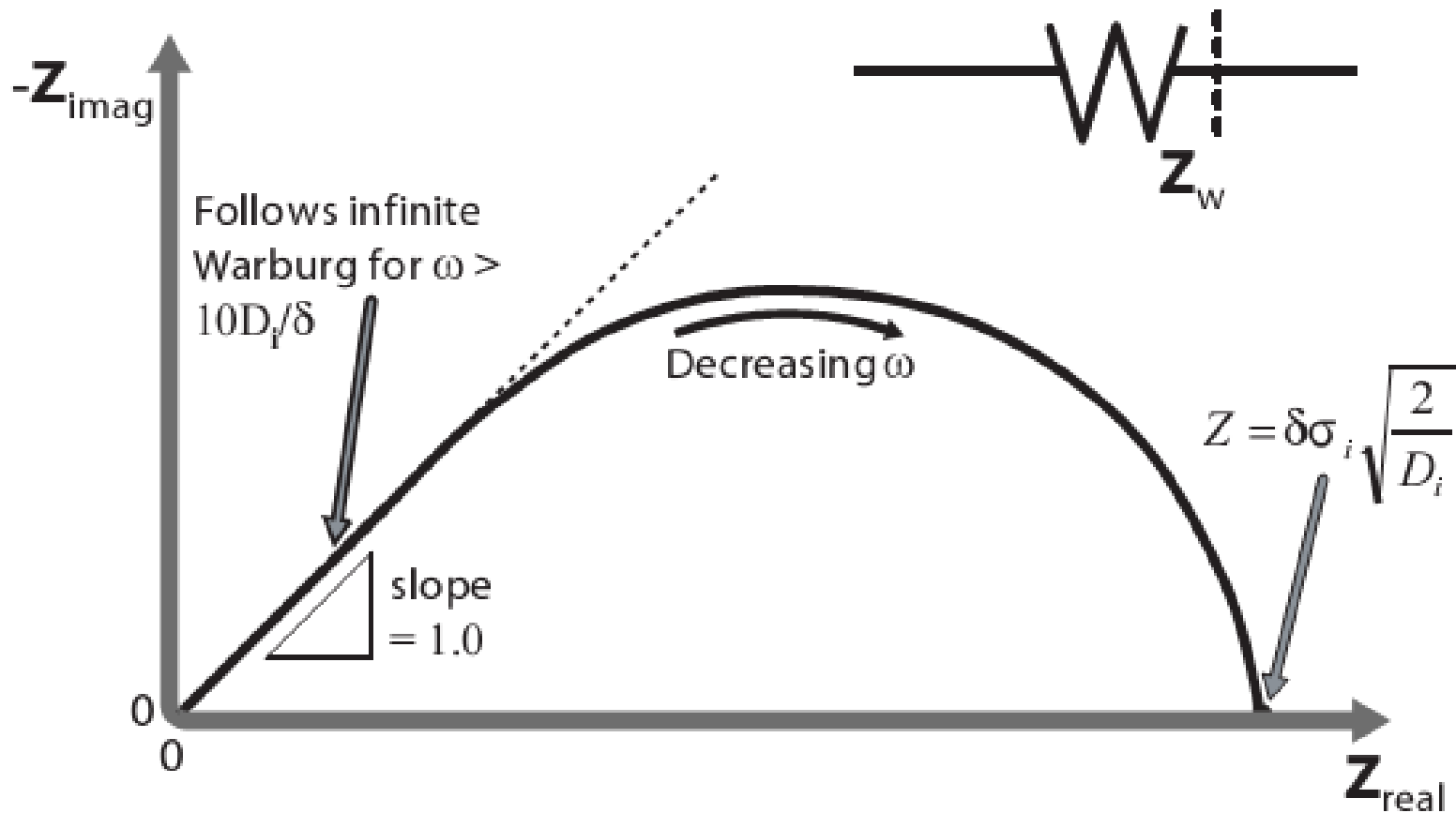
Resistor/Capacitor



Infinite Warburg



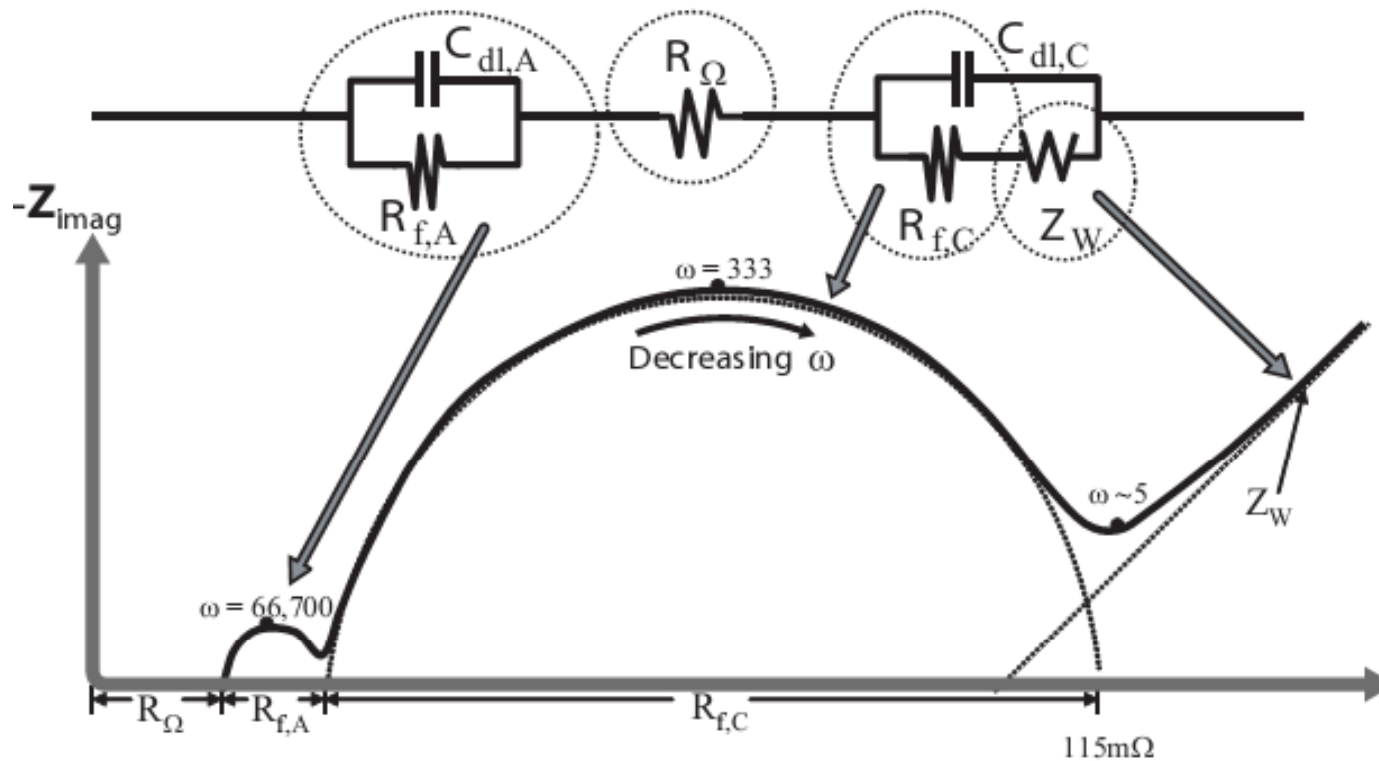
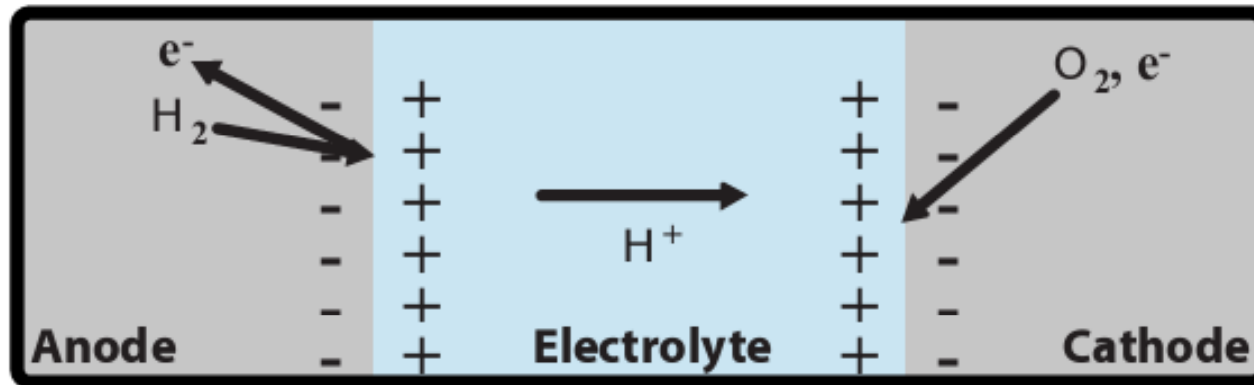
Finite Warburg



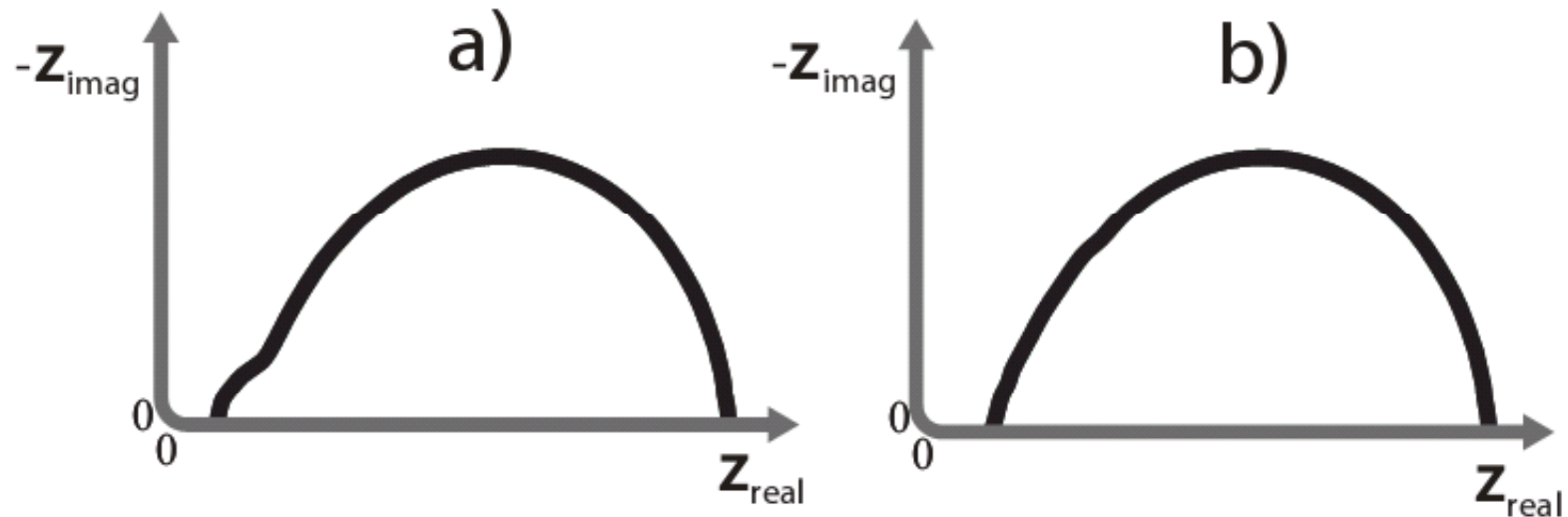
Electrochemical Elements

Circuit Element	Impedance
Resistor	R
Capacitor	$\frac{1}{j\omega C}$
Constant Phase Element	$\frac{1}{(j\omega A)^\alpha}$
Inductor	$j\omega L$
Infinite Warburg	$\frac{\sigma_i}{\sqrt{\omega}}(1 - j)$
Finite (Porous Bounded) Warburg	$\frac{\sigma_i}{\sqrt{\omega}}(1 - j) \tanh\left(\delta \sqrt{\frac{j\omega}{D_i}}\right)$
Series Impedance Elements	$Z_{series} = Z_1 + Z_2$
Parallel Impedance Elements	$\frac{1}{Z_{parallel}} = \frac{1}{Z_1} + \frac{1}{Z_2}$

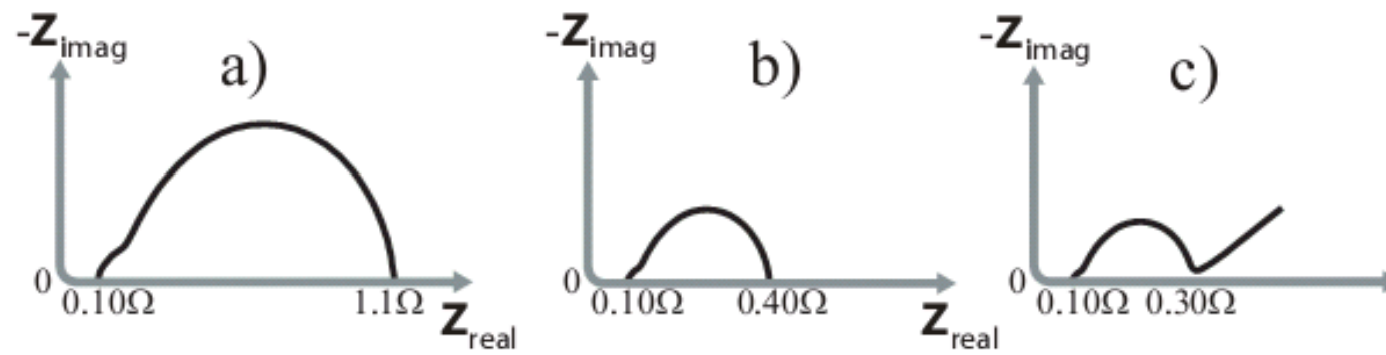
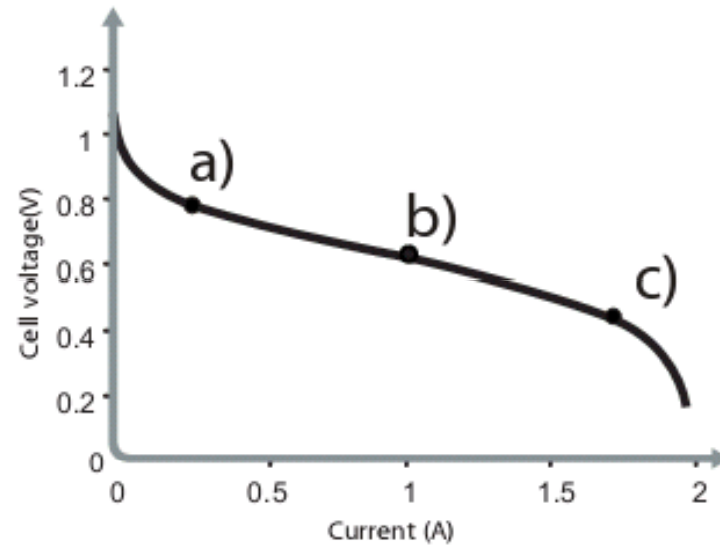
Simple Equivalent Circuit



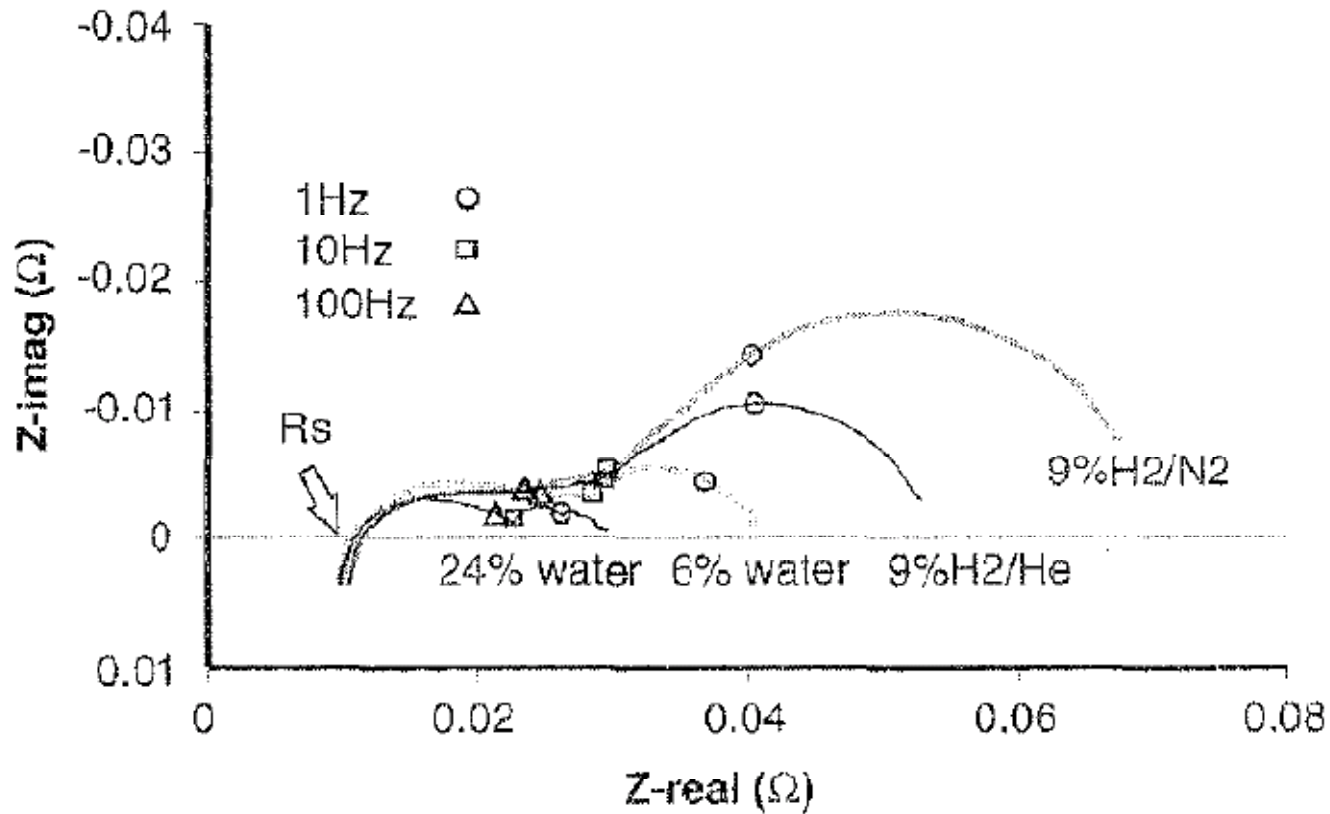
Anode vs Cathode



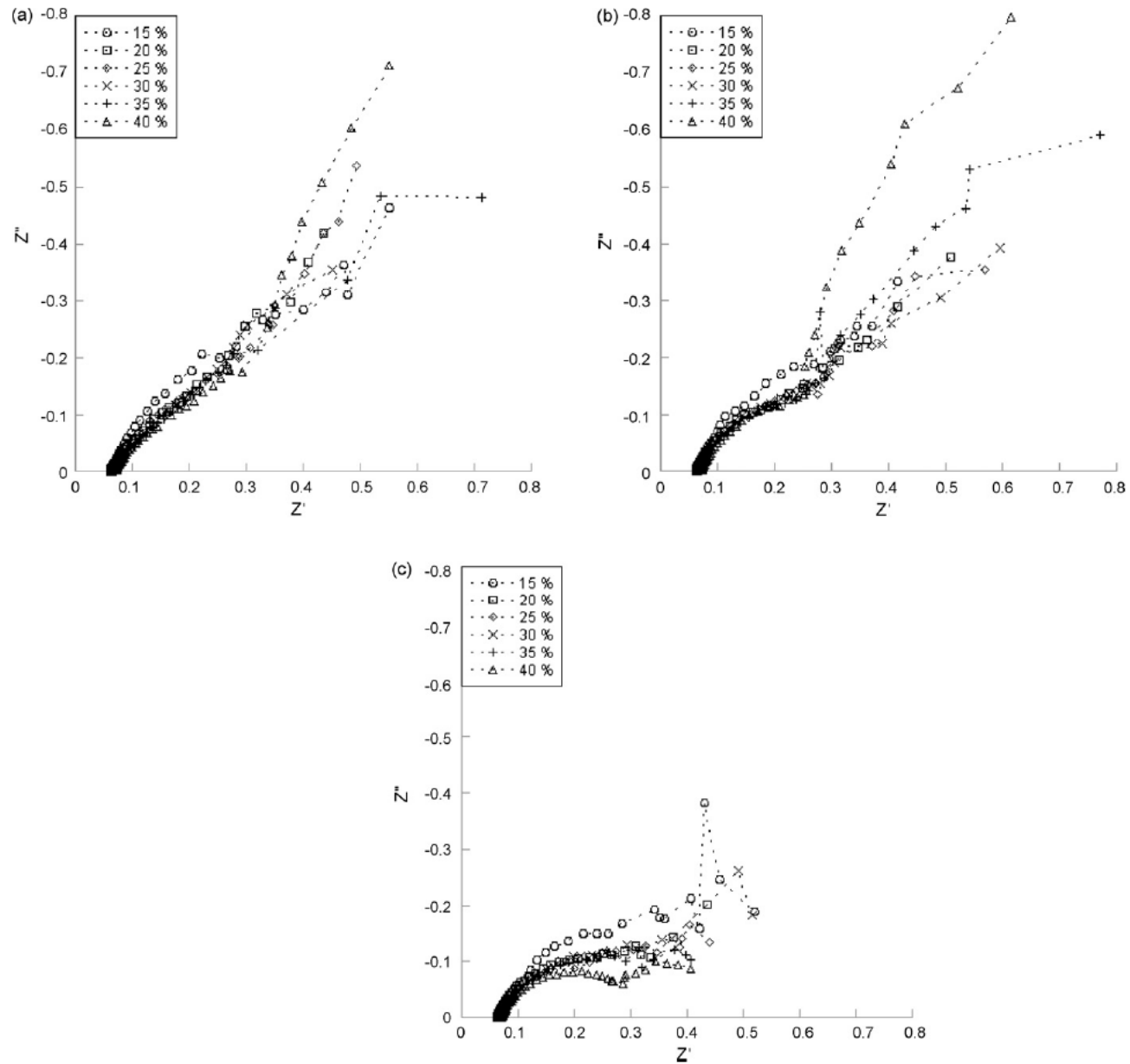
EIS vs Current Density



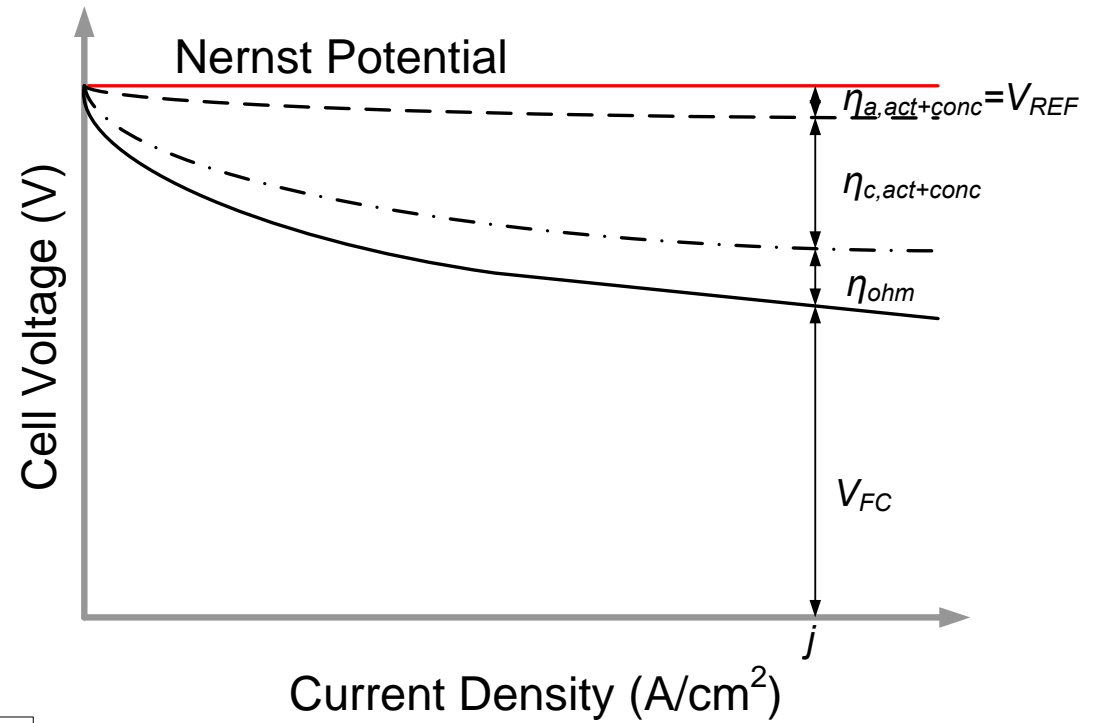
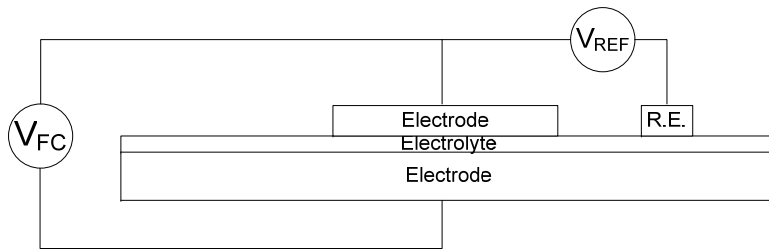
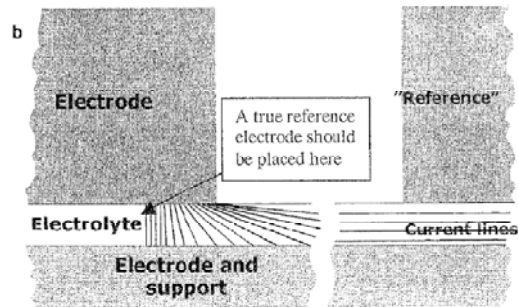
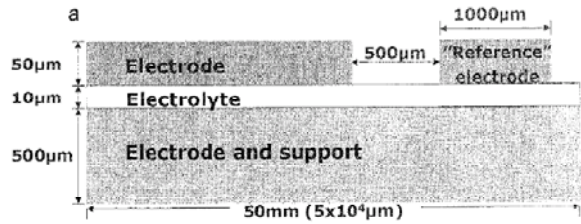
Fuel Composition Effect (SOFC)



DMFC Impedance Example



Reference Electrode



Activation Overvoltage of DMFC

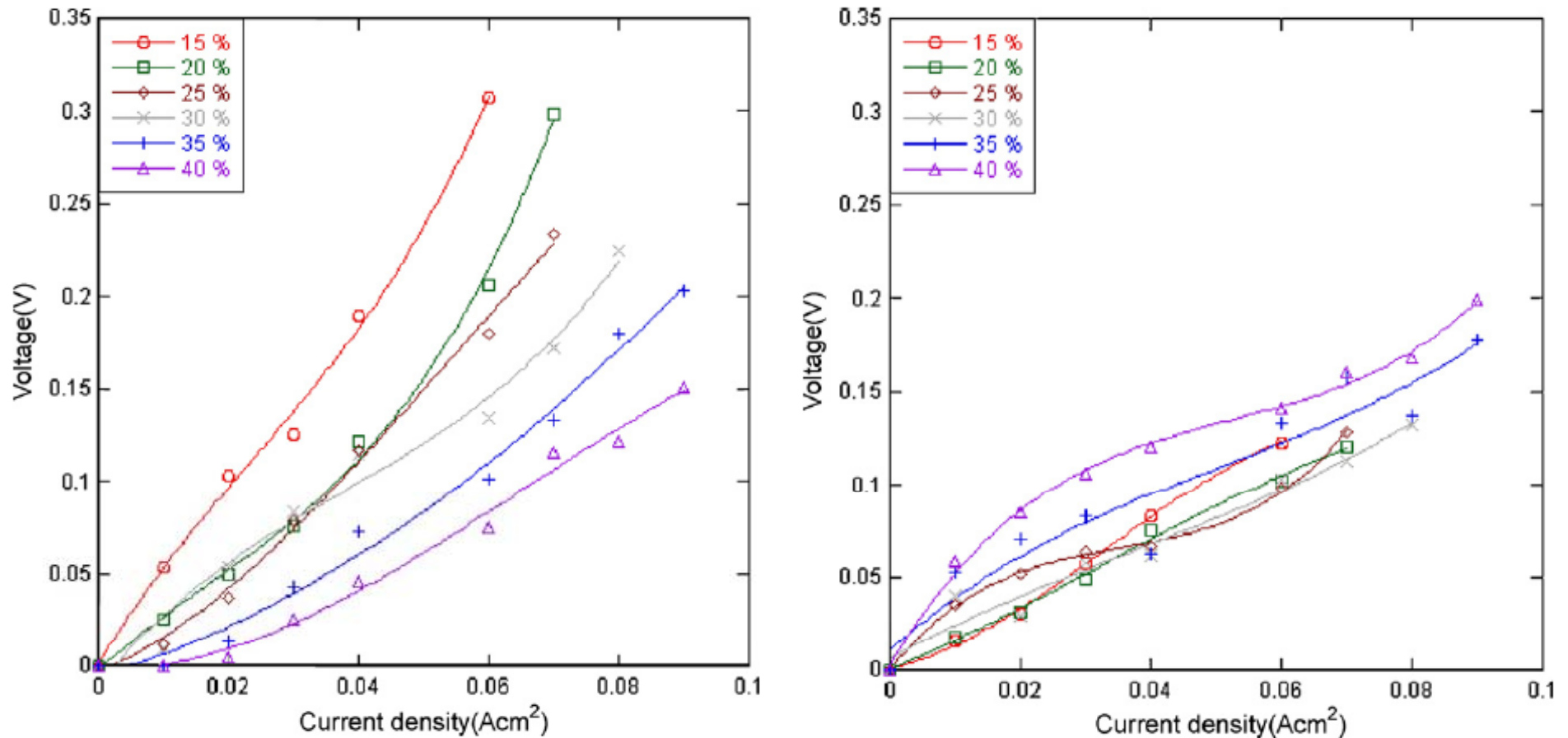
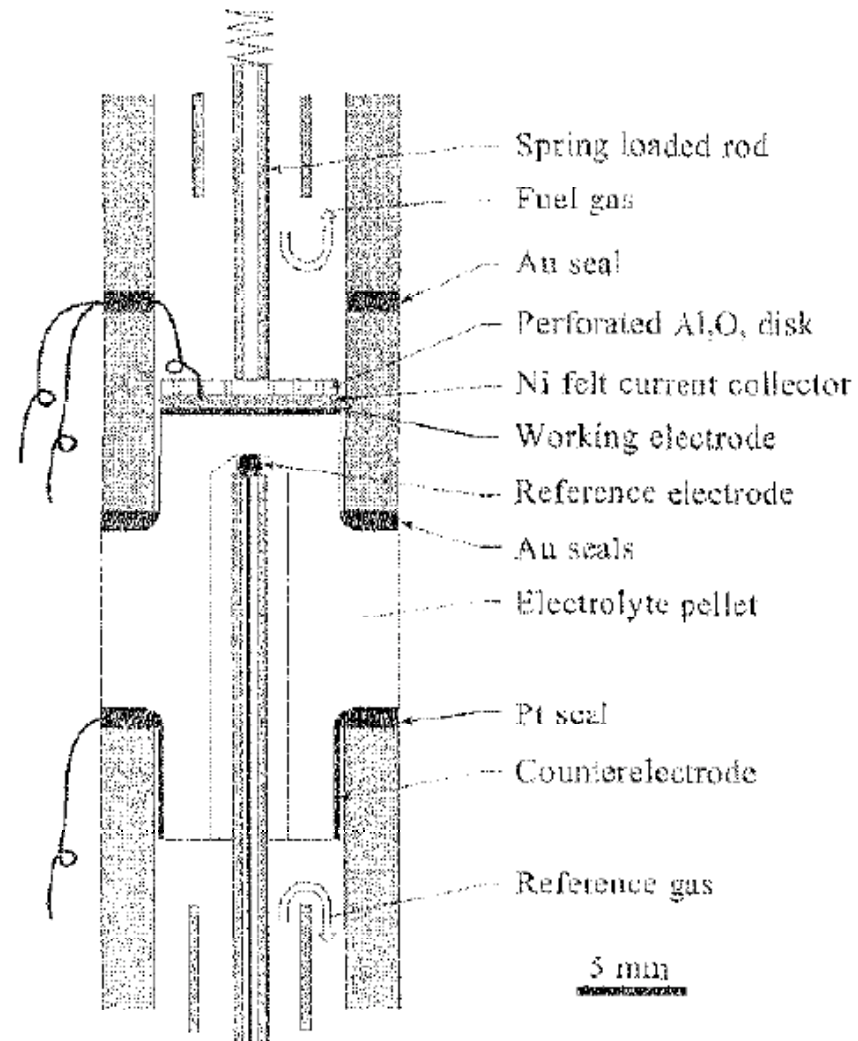


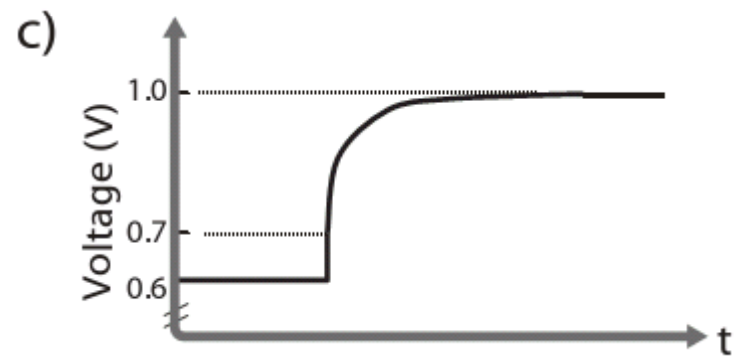
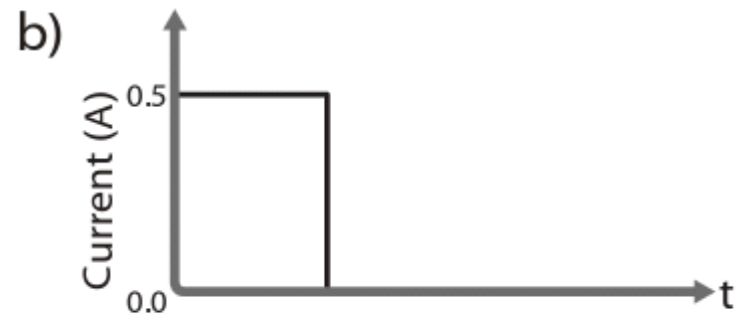
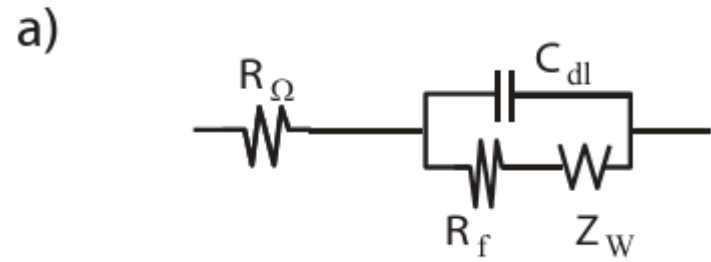
Fig. 9. Activation losses different methanol concentrations at the anode.

SOFC Reference Test Setup

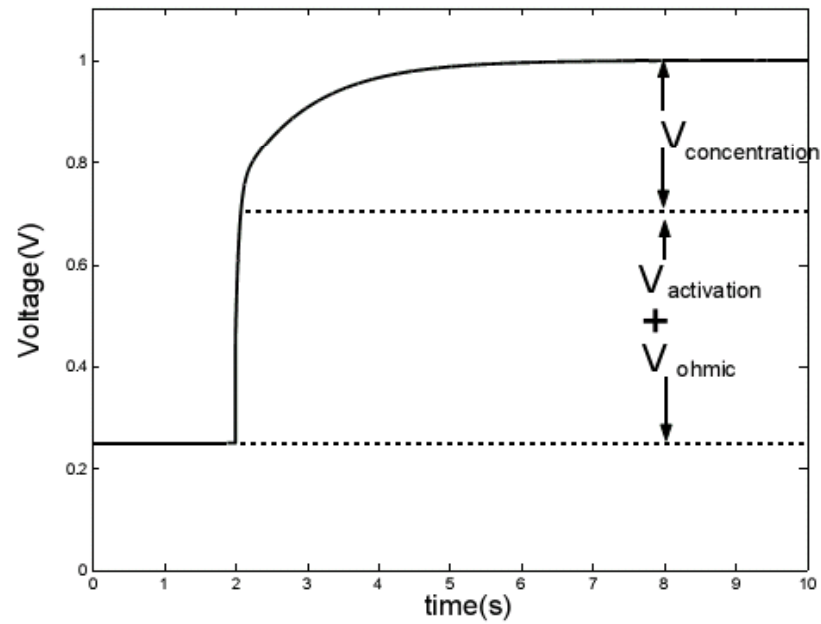


A)

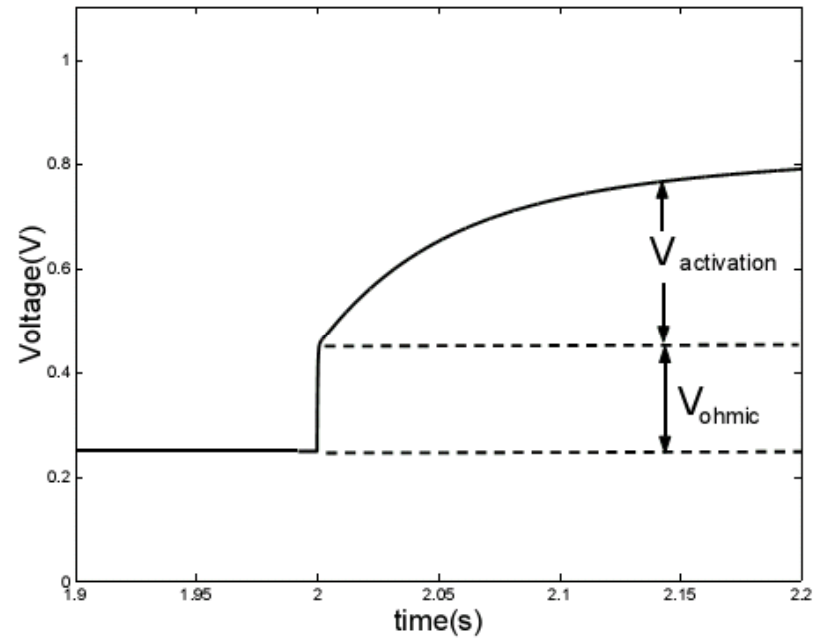
Current Interruption Method



Current Interruption Method

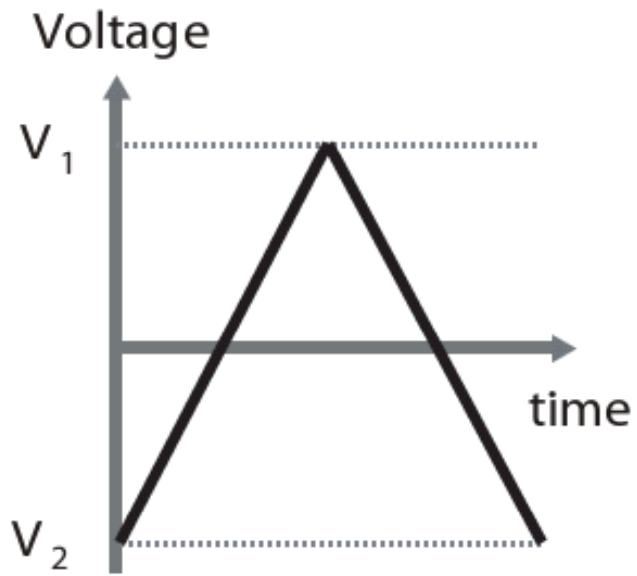


(a)

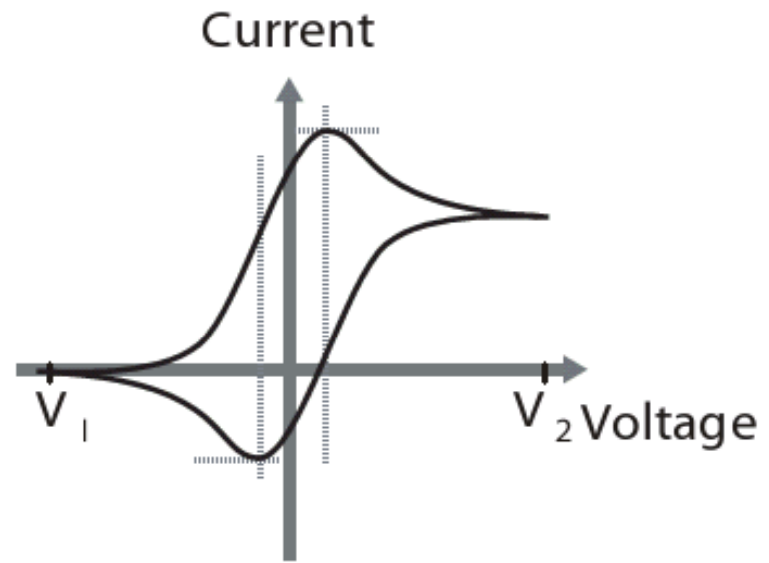


(b)

Cyclic Voltammetry



a) Applied voltage waveform



b) Typical current response

Ex Situ Measurements

- Porosity $\phi = 1 - \frac{\rho_s}{\rho_b}$

– Mercury porosimetry: effective porosity

$$p \geq \frac{2\gamma}{r} \cos \theta$$

- Surface area: B.E.T. method

- Permeability

$$K = \frac{I}{\Delta p} - \frac{\Delta V}{\Delta t} \frac{2p_2}{(p_1 + p_2)\Delta p}$$

- SEM, TEM, AFM, XRD, XPS, FTIR, AES, SIMS, NMR...