Design and Simulation of CMOS Based Potentiostat for Redox Sensor Application

by

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LIST OF ABBREVIATION

- CMRR : Common-mode rejection ratio
- SR : Slew Rate
- PSRR : Power supply rejection ratio
- CM : Common-mode
- PM : Phase Margin
- ARS : Amplified Redox Sensor
- WE : Working electrode
- RE : Reference electrode
- CE : Counter electrode

Reka Bentuk dan Simulasi CMOS Potensiostat untuk kegunaan Sensor Redok

ABSTRAK

Sensor redoks digunakan dengan meluas untuk mengesan analit tertentu dalam larutan kimia, sebagai contoh kepekatan dalam larutan kimia. Sensor redoks mempunyai kelebihan dalam pengesanan pelbagai ion di dalam kawasan sensor dan membuat pengukuran yang ditukar ke voltan atau arus elektrik. Walau bagaimanapun, pengesanan sensor berkurang kerana pengecilan kawasan sensor. Ini menyebabkan keluran isyarat sensor akan kecil. Tesis ini memfokus ke arah reka bentuk potensiostat yang dapat memproses isyarat keluar daripada sensor redoks yang kecil dalam julat 0 μA kepada 10 μA dan mencapai arus voltan yang berganda dan linear untuk diintegrasi dengan Penukar Analog Digital (ADC). Potensiostat direkabentuk daripada tiga blok fungsi yang terdiri daripada blok Sensor, blok Rujukan dan blok Penguat. Blok Sensor dibina untuk memodelkan sensor redoks yang sebenar dan memastikan pengaliran arus elektrik secara linear di terminal keluar. Blok Rujukan akan menjadi bekalan arus elektrik yang tetap kepada dua terminal masuk untuk blok penguat. Blok Penguat direkabentuk kepada penguat pembezaan input dengan output tunggal yang dapat menukar dan menguatkan arus voltan secara linear. Penguat yang direka dapat mencapai DC gain dengan 23.32 dB, PM dengan 90.588º, CM gain dengan -76.019 dB, SR dengan 244.43 V/µs, PSRR dengan 29.41 dB dan CMRR dengan 99.329 dB. Reka bentuk bentangan bagi sistem potentiostat ini dilakukan dan kawasan sel adalah 1719.876 μm^2 dan voltan keluar potensiostat dalam 994.803 mV and 1.4716 9V.

Design and Simulation of CMOS Based Potentiostat for Redox Sensor Application

ABSTRACT

Redox sensor is widely used to detect certain analytes of a chemical solution, for example concentration of a chemical solution. Redox sensor have advantages of detection of multiple ions inside a sensing areas and made measurement and represent it in electrical current or voltage. However when sensing areas is decrease due to miniaturization of sensor, sensor output signal is decrease as well, this is undesired outcome. This thesis will present a design of a potentiostat that able to process the redox sensor small output signal in the range of 0μA to 10μA and have a linear and amplified voltage signal at the output of the potentiostat for integration with ADC circuit. The potentiostat systems contain three functional blocks which made up of Sensor, Reference and Amplifier blocks. Sensor block is constructed to model the real redox sensor and must able to linearly copying current signal to the output. For reference blocks is to act as a constant current sources for two input terminal of the Amplifier blocks. Finally, the Amplifier blocks is designed to be a differential input and single-ended output amplifier which able to linearly convert and amplifier the redox sensor current signal to voltage. The designed amplifier able to achieve DC gain of 23.32dB, phase margin of 90.588º, common-mode gain of -76.019dB, slew rate of 244.43 V/µs, power supply rejection ratio of 29.414dB, and also common-mode rejection ratio of 99.329dB. The layout design for the full potentiostat systems is done with the die areas about $1719.876\mu m^2$ and the potentiostat output voltage is range between 994.803mV and 1.47169V.

Chapter1: Introduction

1.0 Introduction

Electro-chemical sensor is a very useful devices in many scientific field such as in medical science, biological study and also environmental assessment. There are various research on how to use an electro-chemical sensor as redox sensor in chemical analysis to detect oxidation and reduction reaction of chemical species in a solution using a three electrode system that made up of working electrode (WE), reference electrode (RE) and finally a counter electrode(CE).

There are a few advantages of a redox sensor, one of it is the capability to made detection of multiple ions inside a sensing areas and made measurement on the concentration of chemical species in a solution and represent it in electrical current or voltage signal.

However miniaturization of sensor devices for integration with an integrated circuit is indirectly causing sensing areas to decrease and hence causing the sensor output signal to drop tremendously [1]. This small output signal is undesirable outcome as sensing devices have other various application which involve integration with other design that only accept certain range of input voltage such as an ADC circuits. The study on how to made amplification on small current signal of redox sensor output and also the conversion of the current signal to voltages with a potentiostat is an important study for integration with an ADC design.

1.1 Problem Statement

Output signal of an electro-chemical sensor will decrease or getting very small when sensing areas of sensor is decrease due to miniaturization of sensor devices for integration of sensor to an integrated circuit(IC).

A small output signal from a sensor is unfavorable outcomes especially when integration of sensor to analog-to-digital converter (ADC) circuit is an important application as ADC can only take-in a certain range of input voltage.

Boosting electro-chemical sensor small output signal to a readable range for ADC circuit is crucial in the field of electro-chemical sensor study. One of the common ways is to design an amplifier that well integrated with the electrochemical sensor. For sensor output signal in current forms, conversion to voltage is a must as well for integration to ADC as it only have input as voltage.

1.2 Research Work Objectives

The primary objectives of this research works are:

1. To model the redox sensor by constructing a cascode current mirror circuit that able to linearly copying current for redox sensor output signal in the range of 0 μA to 10 μA.
2. To design, simulate and optimize the potentiostat system circuit that made up of a differential amplifier that acts as a trans-impedance amplifier (TIA) to perform current-to-voltage conversion for redox sensor signal and also reference current

circuit that able to output fix and stable current sources to the two input of the differential amplifier design.

3. To perform post-layout simulation for the physical layout design of the proposed potentiostat system.

1.3 Research Work Contribution

A potentiostat systems is design which able to take-in the small current signal from redox sensor in the range from ($0\mu A$ to 10μA) with current-to-voltage conversion of the small current signal at potentiostat output. The small current signal is also able to linearly amplified and observed at output of the potentiostat.

1.4 Scope and Limitation

The scope of this research works is to design a potentiostat systems that can take-in only the small current signal from range of $0\mu A$ to 10μA and able to perform current-to-voltage conversion with a linear output signal observed at potentiostat output terminal. The design is done using silterra 0.18μ process with voltage supply of 1.8V and operates at room temperature of 27^{o}C. After circuit design is done, physical layout design will be perform for the potentiostat system and finally follow by post-layout simulation on potentiostat system output terminal.

1.5 Thesis structure

The thesis is being structured to contain five chapter. The brief explanation of each chapter is as below

Introduction (Chapter 1): This chapter is to give a brief explanation and introduction about the research work that being done for the thesis for electrochemical sensor. The research objectives, contribution as well as limitation will be bring up as well in the chapter

Literature Review (Chapter2): This chapter is a detail review of the field of study of electrochemical sensor with main focus on the electrochemistry of amperometric sensor, operation of a potentiostat, existing published potentiostat topologies, new potentiostat topologies, current mirror design techniques that being used for amperometric sensor, circuit design techniques and concept that being use in Amplifier Redox Sensor(ARS) for chemical solution analysis.

Research Methodology and Implementation (Chapter 3): This chapter will present the method and work flow that being used to come out with the potentiostat design that able to take-in the redox sensor small current signal from $0\mu A$ to 10μA. Detail explanation on the cascaded current mirror design, reference current design and also differential amplifier design that form the full potentiostat system and also physical layout design methodology will be present in this chapter.

Results and discussion (Chapter 4): This chapter will present the results of the cascoded current mirror design, reference current design and also the performance of the differential amplifier that forms the potentiostat systems with post-layout simulation results.

Conclusion and Future Works (Chapter 5): This chapter will made a summary about the data and finding that being present in the previous chapter to come out with the potentiostat design and finally is the future work that can be done for the proposed potentiostat systems.

Chapter2: Literature Review

2.0 Introduction

Chemical sensors is a sensing devices used for detect certain properties of analytes where the analytes can be in the forms of gaseous, liquid or solid. There are varieties types of chemical sensor and they are classified according to the property that they are detecting such as electrical, optical, thermal and other. Electro-chemical sensor is one of the attractive chemical sensor being used among varieties selection of chemical sensor due to its detectability, simplicity and low cost.

Electro-chemical sensors is widely used in many scientific fields such as in biological research, medical science and also environmental study (Takahashi, S., 2014). When the sensors is use to detect certain properties of an analytes, take an example it is detecting the concentration of a chemical solution, a corresponding or proportional output signal will be generated at the sensor outputs. The output signal mostly will take the forms of either voltage or currents.

There are three types of available electro-chemical sensors, they are potentiometric, amperometric and conductometric sensors. For potentiometric sensors, potential difference or voltage between the working electrode and the reference electrode will be measure, the working electrode potential is proportional to the properties of analytes that being detect, for example, potentiometric sensor is measuring concentration of a chemical solution and for conductometric sensors, it involves measurement of conductivity at a series of frequency and finally for amperometric sensor, voltage is applied between references and working electrode, as that it will trigger redox (reduction and oxidation) reaction to occurs, and the resultant current due to the reaction will be measured at sensor output.

Electrochemical sensors will utilizes potentiostat hardware to control the electrode to perform electro-analytical experiments. The three electrode systems made up of three kinds of electrodes is being used. The three electrodes are working electrode (WE), reference electrode (RE) and finally counter electrode (CE) as show in the Figure 2.1(Wang, W.S, 2010).

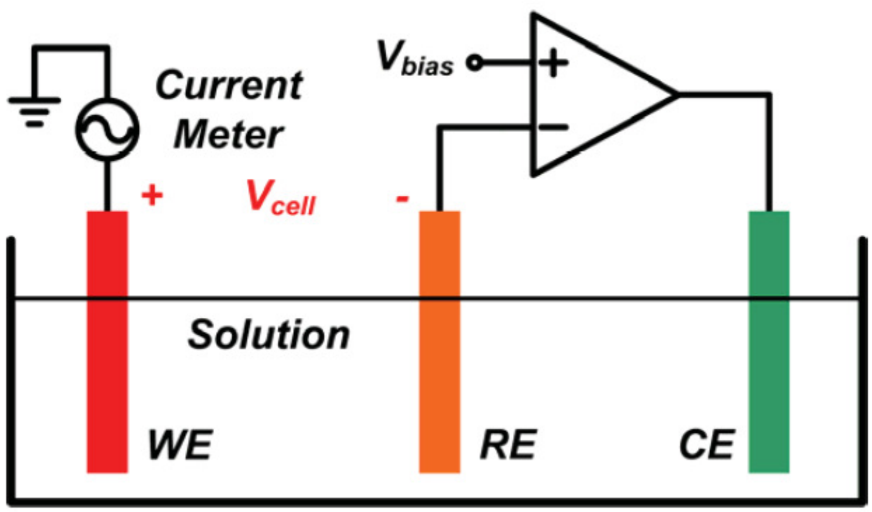

Figure 2.1: Three electrode amperometric electrochemical sensor and potentiostat

Working electrode is where the electro-chemical reaction happens and for reference electrode, its main function is to made measurement for any potential difference present at working electrode.

Reference electrode voltage will be affected by present of current, therefore a high input impedance is required to ensure no current is present at reference electrode, this can be archives by connecting the reference electrode to gate terminal of a MOSFET transistor[3]. For counter electrode, its main function is to serves as a conductor to supply current that required for the electro-chemical reaction to happen at working electrode.

Voltage between WE and RE electrodes will be measured by potentiostat as Figure 2.1 and will be kept in certain value, this happen through the sinking and sourcing of current from and into sensor through CE electrodes(Wang, W.S, 2010), and at the same time the sensor current is being measure.

Electro-chemical sensors is widely used to monitor and detect reduction and oxidation reaction of chemical species. In shorts, electro-chemical sensors is being used as a redox sensors for chemical analysis. Redox sensor have capability to monitor multiples types of ions that present in a sensing areas through detection of the chemical species redox potential (Takahashi, S., 2014), Hence, concentration of ions of a chemical species able to be determine, output signal in the form of voltage or current that proportional to the concentration of a chemical species is generated at the output.

Advancement in nanotechnology have created trends in miniaturization of sensors devices including electro-chemical sensors, thus the sensing areas of sensor is decrease and getting smaller. This cause the corresponding output signal of the sensor to decrease (Takahashi, S., 2014) and is unfavorable to integrate with Analog to digital converter (ADC) circuit as ADC input can only take-in certain range of voltages.

Amplification of output signal of miniaturize electro-chemical sensor with decreasing sensing areas is very important if integration of sensor and ADC circuit is part of the application to be implemented. Very often an operational amplifier circuit will be used to boost up sensor output signal to a readable range for ADC circuits.

There are varieties types of amplifier circuit that able to do amplification for sensor output signal which can range from a single element bipolar junction transistor (BJT) that integrates with sensing electrodes and working electrodes to a more complex systems that integrates trans-impedance amplifier (TIA) circuit with sensor output to perform current to voltage conversion, for example if the application requires sensor devices to integrate with other devices that only take in voltage as inputs.

A lot of work and research have been done on electrical circuit design using CMOS technology in order to integrate semiconductor technology with electro-chemical sensors for wide range of application and field such as in medical treatment, cellular biology and environmental using three electrode systems that mention in the chapter.

In this chapter, focus will be put on discussion of the study of electrochemistry of amperometric sensor, operation of the potentiostat, the current existing potentiostat topologies, new potentiostat topologies such as single ended(SE) potentiostat and fully differential(FD) potentiostat, current mirror circuit design techniques that being implemented for potentiostat, and also discussion on the circuit design techniques and concept for amplifier redox sensor(ARS) for the study of reduction and oxidation reaction of chemical solution.

2.1 Electrochemistry of amperometric sensor

In this section, discussion will be about electrochemistry study on faradaic current, overpotentials and also amperometric sensor

2.1.1Faradaic current

In equilibrium stages where there is no applied voltage, a single polarization electrode in a solution will develop potential based on the chemical species of the solution [16]. When a large voltage which greater than equilibrium potential is applied to the electrode, the systems will be out of initial equilibrium stages, reduction and oxidation reaction occurs which can be explained as below. (Martin, S.M., 2009)

$$O + ne^{-} \Leftrightarrow R \qquad \text{(Equation 2.1)}$$

O is the species oxidized forms, n represent number of electron per molecule being oxidized or reduced, whereas e^{-} represent electron, finally R is the species reduced forms. When reduction and oxidation occurs, electron will be transfer from solution to electrode and vice versa, the resultant faradaic current, I_F can be observed at the electrode surface.

Faradaic current, I_F is a function of oxidized species concentration and the electrode areas, as that faradaic current value represent a specific ion concentration of chemical species (Martin, S.M., 2009).

2.1.2 Overpotentials

In a liquid solution, overpotential is required to trigger redox reaction for a given chemical species, take a cases of insufficient overpotential at electrode, analytical measurement for the chemical species is not possible [16]. To better explain overpotential, another example is a platinum electrode is being use, a liquid solution tends to breakdown for overpotential more positive than 1.5V and more negative than -1.5V, therefore if overpotential is out of the range mention, for example below 1.5V or -1.5V, analytical measurement for the chemical solution is not possible, therefore potentiostat circuit that operate in low voltages have some drawback as certain chemical species might not detectable (Martin, S.M., 2009).

2.1.3 Amperometric Sensor

A conventional or typical amperometric sensor is a three electrode amperometric cell, the three electrode are: working electrode(WE), counter electrode(CE) and finally is the reference electrode(RE) which have being explain earlier as show in Figure 2.2.

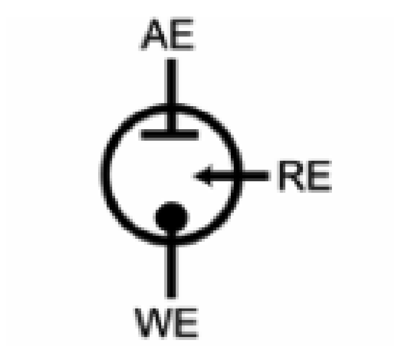

Figure 2.2: Amperometric sensor schematic view

Faradaic reaction of interest will occurs at WE electrodes, and RE electrodes ideally will have zero currents and only for tracking of solution potentials, therefore the potential between electrode and chemical solution which induces faradaic reaction can be given as below

$$V_{WE} - V_{RE} = V_{CELL} \quad \text{(Equation 2.2)}$$

V_{WE} and V_{RE} are potential at WE and RE electrodes, V_{CELL} is amperometric cell potentials. The AE electrodes is use to set potential of solution and sources current for the faradaic reaction (Martin, S.M., 2009).

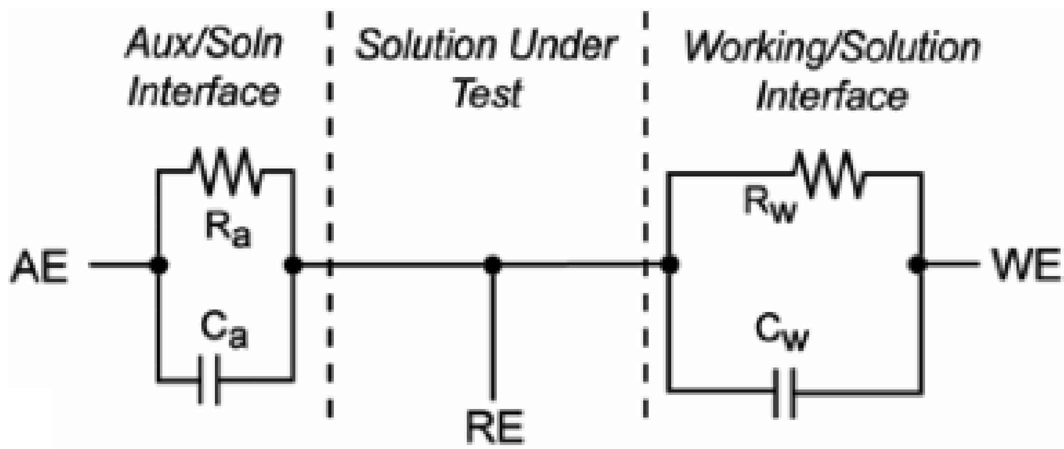

Figure 2.3: Amperometric simplified equivalent model (Ahmadi, M.M 2009)

Electrical-equivalent model of three electrode amperometric cell is show in Figure 2.3. R_A and R_W is representing faradaic resistance, and for C_A and C_W are double layer capacitances associated with AE and WE. The double layer capacitances can be given as

$$C_X = k_C A_X \quad \text{(Equation 2.3)}$$

C_X is electrode capacitance of x, A_X is representing areas of electrode of x, and finally k_C is just a constant value with value approximately 0.36μF/mm^2 and R_W can be defined as

$$R_W \equiv \frac{V_{cell}}{I_F} \qquad \text{(Equation 2.4)}$$

If V_{cell} is having changes, R_W need to be recalculated based on measured faradaic current,I_F but it is inappropriate to assume I_F will increase when V_{cell} is increase as I_F is direct proportional to electrode area, since AE electrode is design to be much larger than WE, therefore $V_{cell} \ll V_{cell}$

2.2 Potentiostat Operation

Figure 2.4(a) shows a WE electrode connected to a voltages sources. No current flows through the metal and solution interface as seen in the ammeter, A as the configuration is missing current return path (Levine 2007).

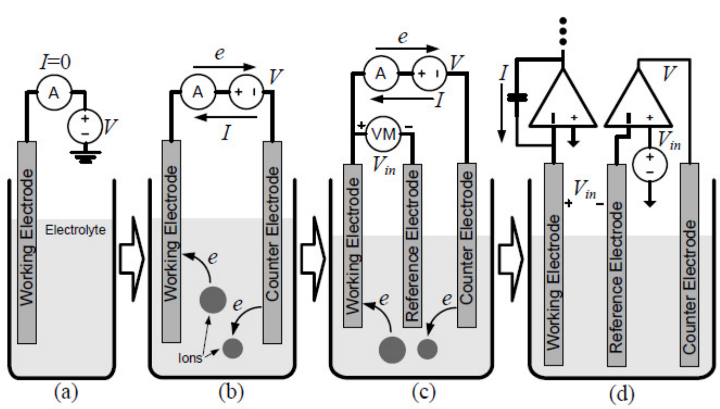

Figure 2.4: Potentiostat operation and circuit implementation

When second electrode, the counter electrode is being placed, there is current flowing as shown in Figure 2.4(b) as ion of the electrolyte is accepting electron and donating electron from one electrode to the other (Levine 2007).

It is hard to distinguish the activity that happen between the WE and CE electrodes as voltage is applied across both of them, therefore a third electrode call reference electrode is added as shown in Figure 2.4(c) to alleviate this issue. The voltage now is vary until a desired voltage level between WE and RE is achieved. A high impedance voltmeter, VM between the WE and RE is to ensure that little or no current is flowing through the RE to maintain equilibrium of the interface. Finally for the Figure 2.4(d), the op-amp at the right side forms a control-loop whereas the integrator on the left is measuring the current..

2.3 Potentiostat Topologies

There are two published potentiostat topologies, they are potential control configuration which potential difference between WE and RE electrode is being controlled and current measurement configuration which current flowing between WE and CE electrodes is being measured (Ahmadi, M.M 2009).

2.3.1 Potential control configurations

Potential of the cell can be controlled in three types of configuration, they are grounded WE electrodes, grounded RE electrodes and finally grounded CE electrodes. Grounded WE electrodes and grounded RE electrodes is identical, thus can be concluded that, there is just two types of configuration (Ahmadi, M.M).

2.3.1.1 Grounded WE electrodes

As shown in the Figure 2.5, grounded WE electrodes configuration, WE is connected to ground and an operational amplifier is controlling the current, I_F to ensure the cell potential is kept at desire preset potential E_i (Ahmadi, M.M 2009).

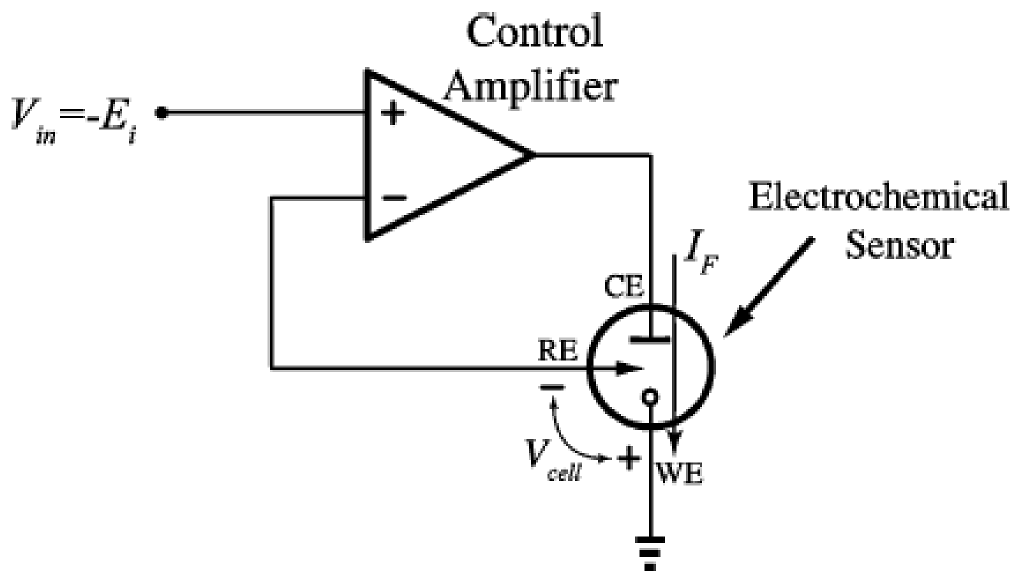

Figure 2.5: Grounded WE electrodes configuration

2.3.1.2 Grounded CE electrodes

This configuration is more complex compared to ground WE electrodes as it require more components, therefore it is more vulnerable to component mismatch as shown in Figure 2.6. For cases which shielding and screening of WE connection from external electromagnetic interference (EMI) are difficult to implement, by using grounded CE configuration have advantage of improving the current measurement .

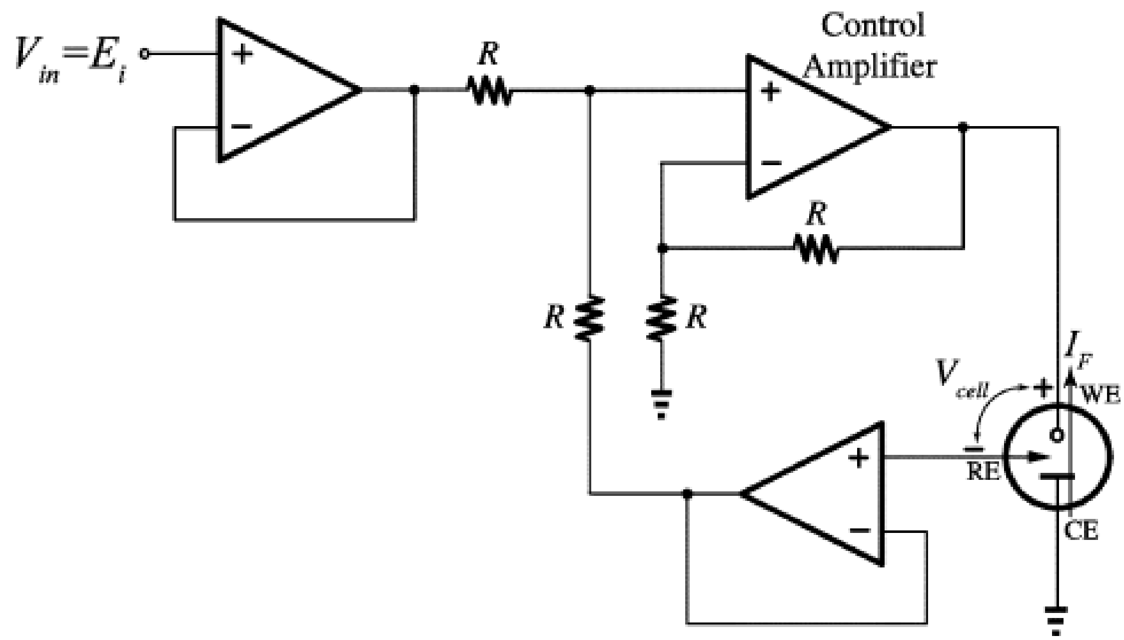

Figure 2.6: Grounded CE electrodes configuration

2.3.2 Current measurement configuration

There are few current measurement configurations, such as current measurement with a transimpedance amplifier, two electrodes electrochemical sensor using current conveyor and current measurement with inserted resistor in current path at WE electrodes.

2.3.2.1Current measurement with a transimpedance amplifier

In this configuration, transimpedance amplifier is being used to force a virtual ground at WE electrodes with an output voltage which linearly proportional to the cell current, I_F as show in Figure 2.7 (Ahmadi, M.M 2009).

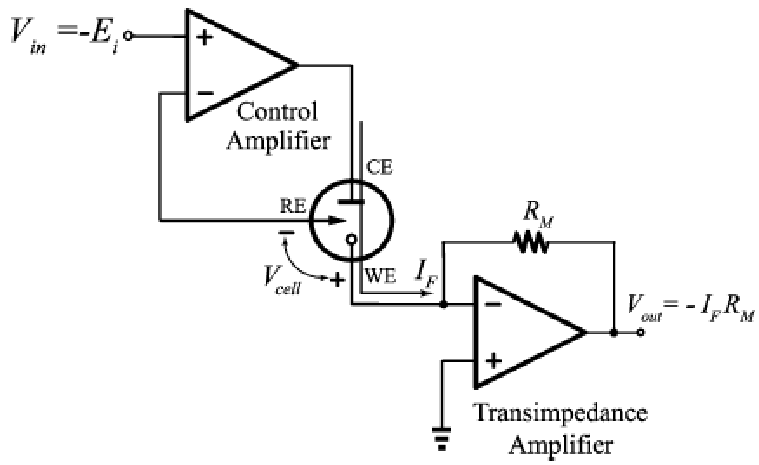

Figure 2.7: Current measurement with transimpedance amplifier configuration

This configuration have advantages on simple to be realized, small current can be measured by swithing the resistor, R_M to higher value and both voltage and current able to be measured with reference to the ground (Ahmadi, M.M 2009).

However, there are three drawbacks on this configuration. The first drawback is the WE electrodes have no true ground connection, and the connection between it and potentiostat is not carefully shielded, thus environment noise and interference will be pick-up, and passed through resistor, R_M, which finally produces noise at the output of transimpedance amplifier (Ahmadi, M.M 2009).

The second drawback is the input resistance of transimpedance amplifier behave inductively, therefore at low frequency, it only exhibit a very small impedance, therefore when frequency is increase, the input impedance will increase, and since input impedance is in series with the electrochemical cell which has large capacitive components, this inductive behaviour of the input impedance has a possibility increase in instability and oscillation in the potential control loop.Finally the third drawback of this configuration is related to single supply voltage and to better understanding of the problem, two types of electrochemical glucose bio-sensor will be used as an example.

There are two types of electrochemical glucose bio-sensors, they are oxygen-electrode-based (O_2 based) and hydrogen-per-oxide-electrode-based (H_2O_2 based) sensor. In O_2 based glucose sensor, cell voltage will be around 600mV(reference to standard Ag/AgCI/ electrode) as shown in Figure 2.8. The reduction reaction of oxygen at WE electrode surface will produce output current,while the RE electrodes is required to maintain at 600mV potential above WE electrodes. Hence, the direction of sensor current is from CE electrodes to WE electrodes, so that output of the transimpedance amplifier must be below the ground potential which is not possible in single supply voltage circuit.

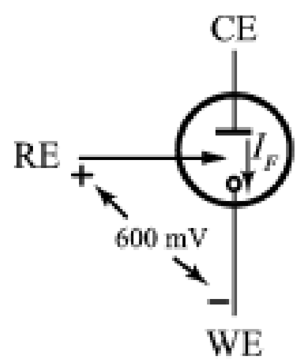

Figure 2.8: O_2 based glucose sensor

As for H_2O_2 based glucose sensor, this situation will be reversed, but the circuit is still facing issues with single supply voltage. Now the sensor current is due to the oxidation reaction of hydrogen peroxide at WE electrodes surface, and current is flowing from WE electrodes to CE electrodes as shown in Figure 2.9. The cell potential of RE electrodes has to be 700mV (Assume Ag/AgCI RE) below potential of WE electrodes which is not possible as well with a single positive supply since WE electrodes is at ground potential.

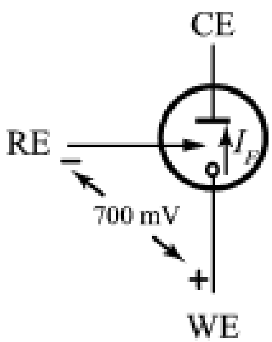

Figure 2.9: H_2O_2 based glucose sensor

2.3.2.2 Two electrodes electrochemical sensor using current conveyor

Current measurement using current conveyor is widely used for two electrode systems as shown in Figure 2.10. For this configuration, WE electrodes will be held at virtual ground potential.

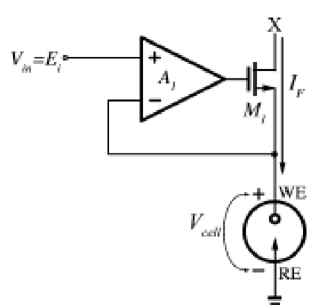

Figure 2.10: Two electrochemical sensor using current converyor

2.3.2.3 Current measurement with inserted resistor in current path at WE electrodes

In this configuration, resistor will be inserted at the current path of WE, the voltage across the resistor can be measured either before or after amplification, there are two approaches for this configuration (Ahmadi, M.M 2009). .

The first approach, WE electrodes will not be ground potential, and the potential changes is depending on I_F. For proper controlling of potential, the potential is measured and fed back to control amplifier as shown in Figure 2.11.

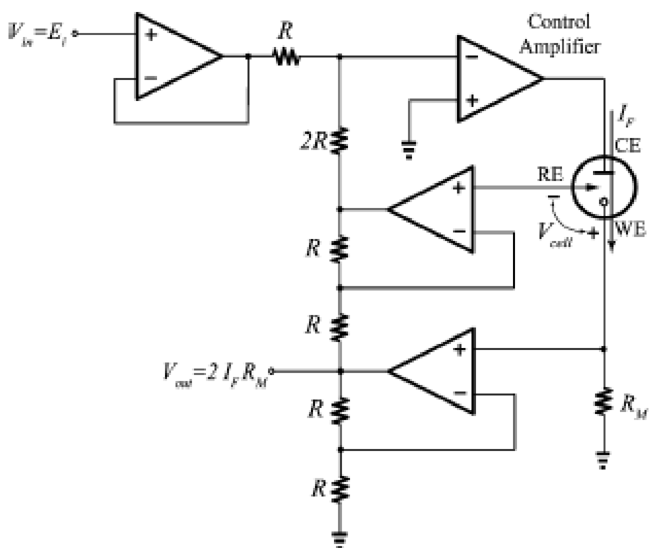

Figure 2.11: Current measurement with inserted resistor in current path at WE electrodes

This configuration is more complex comparing of using transimpedance amplifier as it require more active as well as passive components. This result in the circuit that have move noise and more vulnerable to mismatch of components but the circuit is still have good capability of measuring very small currents. Both the current and voltage measurement of the circuit with reference to the ground.

The second approach, current measurement involve inserting of resistor at CE electrodes current path and followed by measurement of voltage across the resistor as shown in Figure 2.12. This configuration is facing the same problem on mismatches of component that was discuss earlier but have few advantages(Ahmadi, M.M 2009).

The first advantage is WE have true ground connection, therefore very insensitive to noise and interference pickup. The second advantage is there is no additonal active component in the control feedback loop(excluded control amplifier), the circuit will have better stability compare to other. Finally the third advantage is both current and voltage can be measured with reference to the ground (Ahmadi, M.M 2009)..

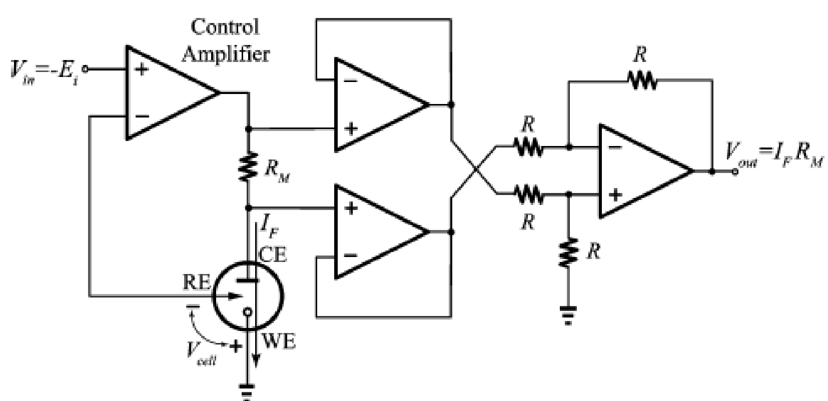

Figure 2.12: Current measurement insert resistor at CE electrodes current path

The last two configuration that have been discussed do not have issues related to the single positive voltage in first configuration, but the last two configuration is suffering from another problem, that is when sensor current increase, the voltage drop across resistor R_M increase, this cause decrease of voltage swing at CE electrodes.

2.4 Amplifier Redox Sensor(ARS) for chemical analysis

Redox sensor from the name itself imply a sensor which detect reduction and oxidation reaction and has wide application as mention in the early stages of the chapter, one example is study of the sensor for imaging cell activity and monitoring of environment pollution (Takahashi, S. 2014).

Redox sensor can be implemented using a bipolar transistor combined with three electrodes systems. This configuration allow amplification of a very small current signal and also measurement of both positive and negative currents. Another type of configuration been proposed consists of an additional diode than just a single bipolar transistor.

2.4.1 Basic Redox Sensor without BJT

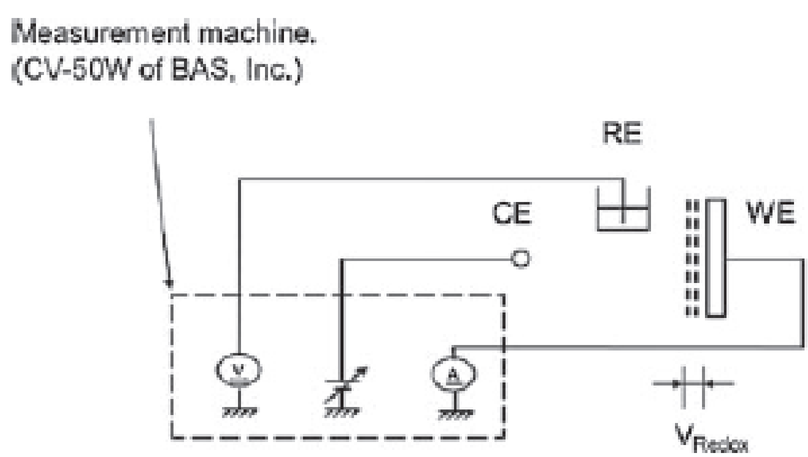

Figure 2.13: Basic measurement systems for redox sensor

Figure 2.13 show a redox sensor basic measurement systems. Three electrode system is being used to measure the redox current and potential. WE is for current measurement, CE is to apply voltage to the chemical solution and finally the RE is to measure the voltage of the solution (Takahashi, S. 2014)..

Figure show the results of current and voltage measurement using BAS CV-50W. The chemical solution concentration is taking the absolute value of peak current and for redox potential, it is taking midpoint of potential of reduction and oxidation peak current. From figure 2.14, peak positive and negative current can be measure and observed

Each chemical species or substance will have own redox potential, therefore identification of a specific chemical species can be archives by knowing the redox potential. The redox current that flowing at WE can be express as:

$$I_p = (2.69 \text{ x } 10^5) n^{3/2} \mathrm{A} D^{1/2} \mathrm{C} v^{1/2}$$

I_p is peak current, n is number of electrons, A is the WE areas, D is diffusion constant, v is scan rate, C is bulk concentration, therefore from the equation, can concluded that sensor current is linearly depend on sensing area sizes and also ion concentration.

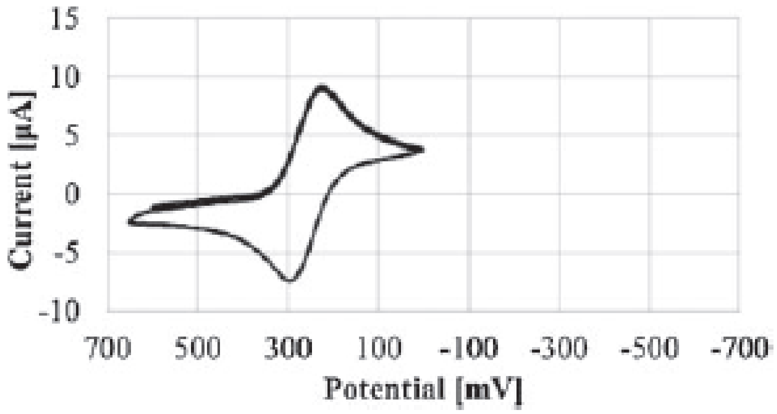

Figure 2.14: Current and voltage measurement of redox sensors

2.4.2 Redox sensor with BJT for amplification

As show in Figure 2.15, WE is connected to the BJT base terminal, a bias voltage is applied to the collector terminal, the amplified current at emitter terminal can be observed and redox voltage will be shift also due to present of forward voltage of bipolar transistor (Takahashi, S. 2014).

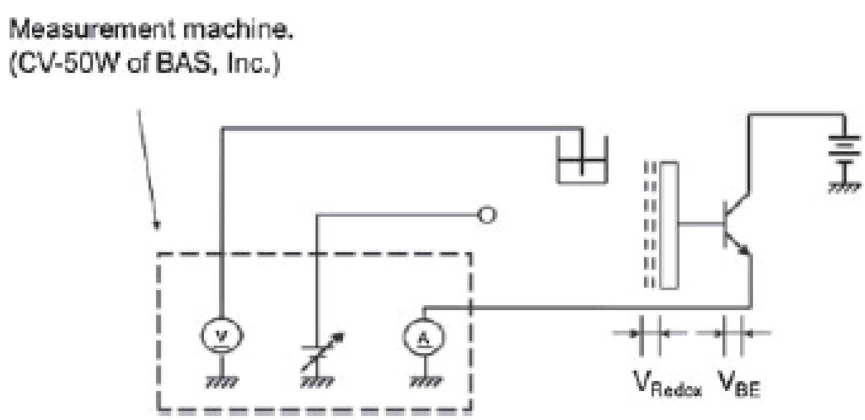

Figure 2.15: Redox sensor with BJT