IPS Journal of Physical Sciences
ISSN: 3115-5715
IPS J. Phys. Sci., 3(1): 155-159 (2026)
DOI: https://doi.org/10.54117/ijps.v3i1.28

Development of Arduino-Based Cyclic Voltammetry Analyzer for Semiconductor Growth

O. I. Olusola*, T. I. Ogunniyi, T. A. Obagade, K. D. Adedayo, T. Ewetumo

Department of Physics, School of Physical Sciences, The Federal University of Technology Akure, Nigeria

*Corresponding author- oiolusola@futa.edu.ng / olajideibk@yahoo.com

Abstract

This paper presents the development of a three-wired probe potentiostat for use in growing semiconductor thin film materials. The cyclic voltammetry analyzer is a potentiostat designed for the purpose of performing cyclic voltammograms and thin films growth. The cyclic voltammetry analyzer was developed using an Arduino mega microcontroller unit, a 12-bit analog to digital converter, a 4x4 matrix keyboard, a digital liquid crystal display unit, and a data logger. Sets of probes were used in connecting the working electrode and counter electrodes inserted in the prepared electrolytic bath containing the various ions to be deposited to the potentiostat which controls the supplied current to the electrode. The microcontroller was programmed using Arduino C code to apply the potential sweep and record the current response for the cyclic voltammogram of the sample (working electrode). The cyclic voltammograms of zinc sulfide (ZnS), cadmium sulfide (CdS), and aluminum gallium selenide (AlGaSe) were analyzed using current and voltage data from the developed instrument to determine the suitable potential range for electrodepositing the semiconductor. The results obtained in this work showed compatibility with the obtainable results in the literature.

Keywords: Cyclic Voltammetry Analyzer, Potentiostat, Arduino - based Microcontroller, Semiconductors, Potential.

Article History
Received: 30 Jan 2026
Accepted: 09 Mar 2026
Published: 17 May 2026

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How to cite this paper: Olusola, O. I., Ogunniyi, T. I., Obagade, T. A., Adedayo, K. D., & Ewetumo, T. (2026). Development of Arduino-Based Cyclic Voltammetry Analyzer for Semiconductor Growth. *IPS Journal of Physical Sciences*, *3*(1), 155–159. https://doi.org/10.54117/ijps.v3i1.28

1. Introduction

The combined effects of electricity and the chemical reaction that takes place at the interface between an electrolyte and the working electrode are studied using potentiostats in a variety of electrochemical analytical techniques. The potentiostat applications include the development and characterization of semiconductors, gas sensors, environmental monitoring equipment, assessing the thermodynamic kinetic parameters of electron transport, and researching the characteristics of supercapacitors [2]. There are essentially two types of potentiostats: the general-purpose potentiostat, which researchers use to perform different types of voltammetry techniques in the laboratory, and the special-purpose potentiostat, which is made for a particular type of voltammetry technique that is appropriate for the research being conducted. The former is costly, complicated in design, and less portable, whereas the latter has the advantages of being inexpensive, easy to use, less power consumption, and portability. The current work falls into the latter category, meaning that it is more effective in determining the suitable potential for semiconductor growth. Aremo et al., (2015) also reported that challenges posed by the general purpose potentiostat has compelled researchers to compromise the specific objective of their research work to the capacity of the special-purpose laboratory potentiostat [2].

The cyclic voltammetry is a dominant tool to study the electrochemical process of a system by methodical study of current-voltage (I-V) measurements of a given electrochemical cell [5]. According to [4], a cyclic voltammetry (CV) analyzer is an electrochemical instrument that linearly varies the applied potential at the working electrode of a three-electrode cell in the forward and reverse directions. This technique, known as cyclic voltammetry, is a potentiodynamic process that sweeps the potential at a predetermined rate while measuring the resulting current. A single cycle typically runs from the initial potential to a switching potential, then back to the initial potential, though the range can be adjusted based on the experiment.

Voltammetry analyzers can use either a two-electrode or three-electrode cell system. In a three-electrode setup, the potentiostat maintains a constant potential between the working electrode (WE) and the reference electrode (RE) by adjusting the current through the counter electrode (CE). The potential difference between WE and CE is not fixed and varies as needed to sustain the desired WE-RE potential. In a two-electrode system, the potential is applied directly between the WE and CE. [10]

Electroplating the various semiconductor materials and studying their individual properties require an essential determination of the range of cathodic potentials suitable for their deposition [3]. Also, the study of redox processes, stability of reaction products and understanding reaction intermediate has made cyclic voltammetry an indispensable and widely used electrochemical techniques in modern electrochemistry [7]. Depending on the analysis, electrochemical analysis may call for a partial, complete, or even multiple cycle. The primary factors required to run the CV analyzer are the voltage range and the scan rate.

The demand for electrochemical workstation in a developing nation, like Nigeria make it necessary for the development of this device. Also, the available potentiostats in the scientific markets are very costly and difficult for individual researchers to afford considering the dollars to naira exchange rate, especially in developing nations. For this reason, the current work outlines the development of a portable, low-cost cyclic voltammetry analyzer for semiconductor growth that is based on an Arduino framework.

2. Materials and Methods

The system architecture of the developed device is illustrated in the block diagram depicted in Figure 1. The device consists of the following components: (a) A digital-to-analog converter (DAC) which generates the setpoint voltage for the control amplifier input, and digital control signals from the microcontroller that drive the relay module; (b) A potentiostat circuit which includes a control amplifier that maintains the desired potential between the working electrode (WE) and reference electrode (RE) by adjusting the current flowing through the counter electrode (CE); the second stage of the potentiostat is the current measuring circuit; that is, a current-to-voltage converter (transimpedance amplifier) which converts the electrode current into measurable voltage while the potential is being maintained; (c) An Arduino Mega 2560 microcontroller board, which is the core controller of the device, processes the measured voltage/current and controls the modulated waveform generated by the DAC. (d) A 4x4 matrix keypad and push buttons serve as the main input devices for setting the voltage range and scan rate of the device. Also, the data logger is integrated into the device to store the measured data for further analysis, while the 16x2 LCD displays the input parameters, measured voltage, and current. The complete system was powered using two separate voltage levels, ±9 V and ±5 V.

The microcontroller's activity is controlled by an embedded C program that is uploaded into it using an Arduino IDE software since it is the easiest one to learn, A 12-bit ADC was used to convert the measured analogue voltage to digital form for better control and data conversion between the potentiostat and computer system [6].

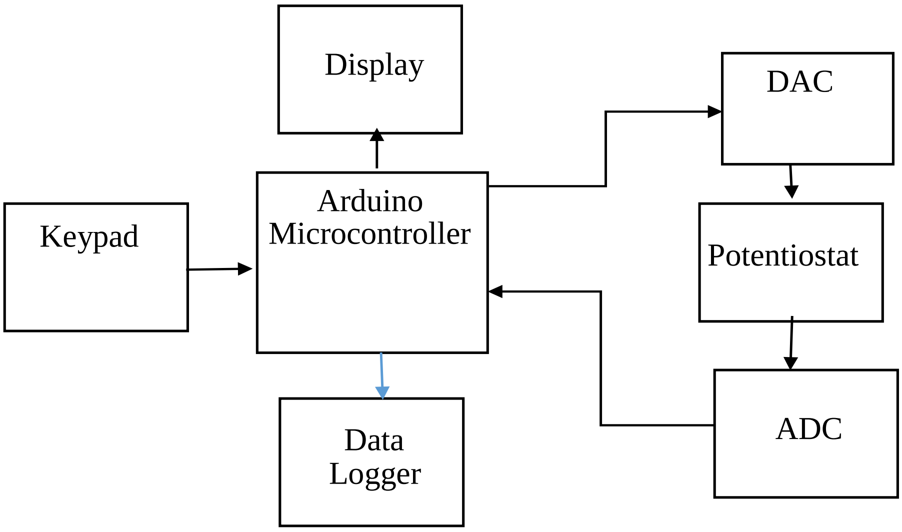

Figure 1: Basic block diagram of cyclic voltammetry analyzer.

Since Arduino lacks a true DAC, an external 12-bit DAC (MCP4725), op-amps (TL084 and TL082), a relay module, and other components were employed for precise voltage ramping. The DAC's output is connected to the input of two op-amps that are configured as buffer amplifiers and a unity gain inverting amplifier. The buffer amplifier's output (A) provides a positive potential to the control amplifier, while the inverting amplifier's output (B) provides the negative potential (See Figure 2 and 3). This was accomplished by using a relay module to switch between the outputs of these operational amplifiers, as illustrated in Figure 2. This signal is tied to a buffer amplifier that prevents the DAC from being loaded. The reference electrode is connected to a high-impedance voltage follower amplifier (D) to prevent current from flowing through it. A transimpedance amplifier connected to the working electrode measures the current flowing through the working electrode as shown in Figure 3. A 0.1µF capacitor connected in parallel with its feedback resistor aids in maintaining the stability of the potentiostat.

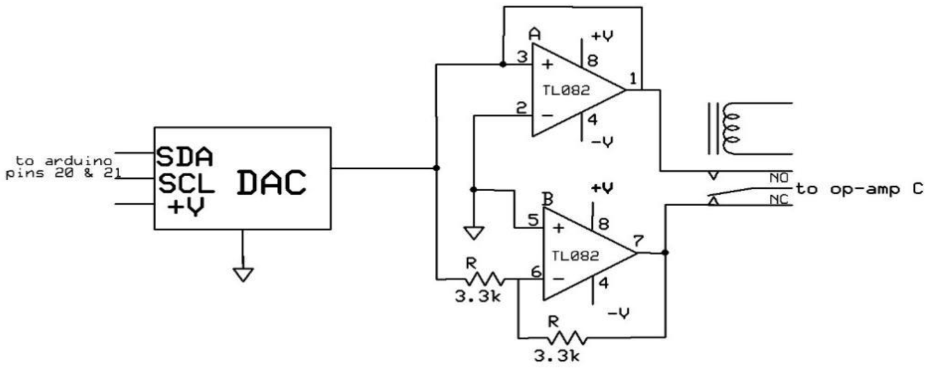

Figure 2: Analogue voltage generator circuit.

Equation (1) gives the resolution of the 12-bit ADC which is about 7.63 GμV [2].

$$Resolution = \frac{GV_{ref}}{n^2} \quad (1)$$

where G is the ADC's gain, n is the number of bits, and the applied voltage, V_i at the virtual ground, is subtracted from the feedback voltage, V_f, using Equation (2). Ohm's law which is explained by Equation (3) was used to determine the actual current flowing through the circuit [2].

$$V = V_f - V_i \quad (2)$$

$$V = IR_F \quad (3)$$

where $V_i = 2.5$ V, R_F is the feedback resistor of the trans-impedance amplifier and V, is the output voltage.

The voltage at the non-inverting input can be obtained using the voltage divider rule, and the non-inverting gain equation is used to calculate the output voltage, V_{out} as given in Equation (4) [9].

$$V_{out} = \frac{R_4}{R_3}\,(V_{out} - V_{in}) \quad (4)$$

As indicated in Figure 3, a 16x2 Hitachi's HD44780 liquid crystal display (LCD) module was used to display the measured current and voltage during the experiment. The LCD was connected to the microcontroller via an I^2C interface, which requires only two lines: SDA for data and SCL for clock, as shown in Figure 3 [11]. A data logger was also incorporated into the device to store the measured data for offline analysis.

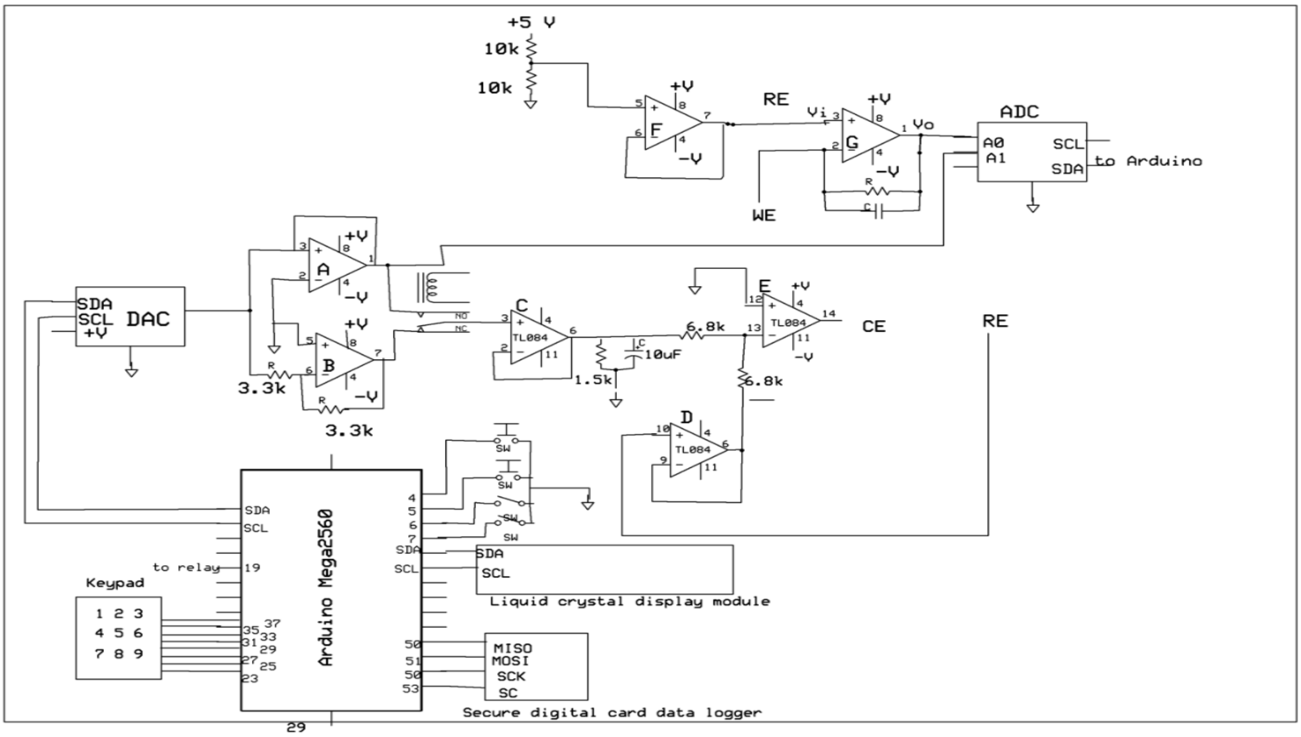

Figure 3: Complete circuit diagram of the cyclic voltammetry analyzer.

3. Experimental Set-Up and Results

3.1 Cyclic Voltammetry of Cadmium Sulphide (CdS)

The developed device was setup to obtain the cyclic voltammogram of CdS thin films. A potential range of -2500 to +2500 mV was applied to the working electrode at a sweep rate of 100 mV/s. Two salts namely, $3CdSO_4.8H_2O$ and $Na_2S_2O_3.5H_2O$, were used to prepare the cadmium sulphide electrolytic bath used for the cyclic voltammetry. The solution was agitated on a magnetic stirrer to ensure the salts would dissolve properly in the solvent. An effective way to identify the appropriate potential range at which CdS deposition and dissolution take place is to look at the electrolyte's I-V curve in both forward and reverse orientations. As depicted in Figure 4, the forward current increased steadily. When the curve initially crosses the x-axis at about 900 mV, it indicates the cathodic potential at which the binary compound CdS begins to deposit on the fluorine-doped tin oxide (FTO) conducting glass electrode. From this point, the current increases gradually to the peak potential at about 1480 mV. The potential range at this area of steady growth in current shows the proper potentials at which CdS can be grown on the electrode. To ascertain the true potential to deposit stoichiometry cadmium sulfide, it is therefore safe to investigate the 900–1500 mV potential range. The voltage range found in this study is consistent with [10] findings, which showed that CdS thin films could be grown at voltages between 900 and 1500 mV. The following is the chemical reaction that occurs when CdS is deposited on the working electrode:

$$Cd^{2} + S_2O_3^{2-} + 2e^{-} \rightarrow CdS + SO_3^{2-} \qquad (5)$$

3.2 Cyclic Voltammetry of Zinc Sulphide (ZnS)

A linear potential scan from 0 to 2000 mV was applied between the working and counter electrodes at a scan rate of 100 mV/s in a two-electrode system. It should be noted that a three-electrode potentiostat was developed as reported in this work. However, while performing the voltammetry analysis, two electrodes (WE and CE) were connected to the two probes of potentiostat designated as WE and CE probes. The third probe which was supposed to be connected to reference electrode was short-circuited with the CE. The reference electrode was not used in this research due to previous report by Dharmadasa (2013) about the possibility of ions such as Ag^{+} which are present in Ag/AgCl reference electrode, leaking into the electrolytic bath that contains the ions of interest to be electrodeposited. This leakage has been reported to act as unwanted dopants which is detrimental to the optoelectronic properties of semiconductor materials [3].

During the cathodic sweep, the FTO working electrode was made negative relative to the carbon counter electrode, driving reduction at the FTO surface. The electrolyte contained 0.2 M of $ZnSO_4.7H_2O$ and 0.02 M of $Na_2S_2O_3.5H_2O$ which act as the sources of Zn and S respectively. The cyclic voltammogram is displayed in Figure 5. In contrast to zinc, which has a negative redox potential, sulfur has a positive redox potential and will therefore probably begin to deposit at a low potential [1]. Considering the forward cycle, current was reported to be constant starting from ~1100 mV. This point represents the initial potential at which ZnS starts to deposit on the FTO glass. Up to 1600 mV, the current remains almost consistent. The deposition of a Zn-rich ZnS coating, which can result in dendritic development on the electrode surface, is indicated by the decrease in current as the potential rises for potentials higher than 1600 mV. As seen in this work, the current is somewhat constant at the potential range of 1100 to 1600 mV, indicating the potential to investigate in order to determine the stoichiometry growth of ZnS thin films. This potential range is consistent with the findings published by [8]. The potential window for electrodepositing ZnS thin films was determined from the cyclic voltammetry in Figure 5 and is summarized in Table 1.

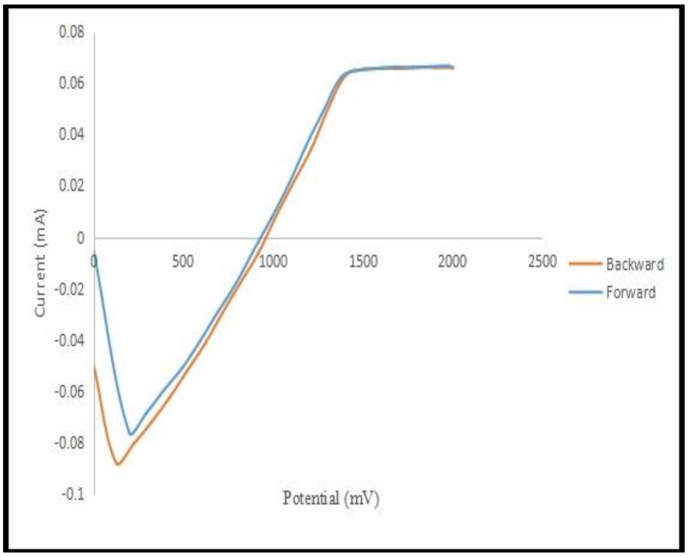

Figure 4: Cyclic voltammogram of cadmium sulphide using scan rate of 100 mV/s.

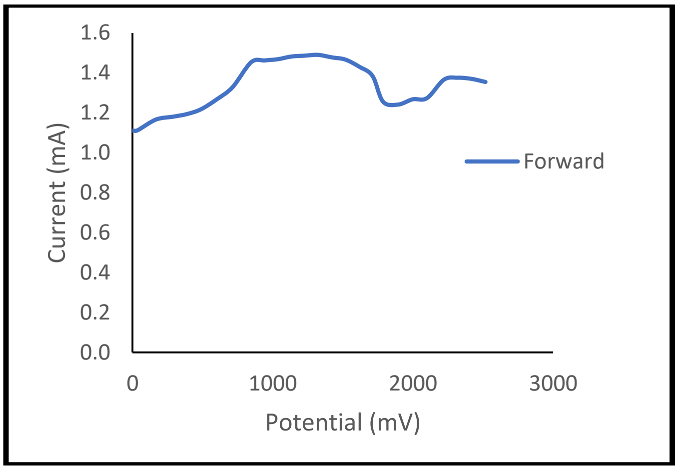

Figure 5: Cyclic voltammogram of zinc sulphide using scan rate of 50 mV/s.

3.3 Cyclic Voltammetry of Aluminum Gallium Selenide (AlGaSe)

Aluminum gallium selenide (AlGaSe) is a ternary semiconductor, and the I-V curve was obtained using the developed instrument. The electrolytic bath used for the cyclic voltammetry was made up of three salts: aluminum chloride hexahydrate ($AlCl_3.6H_2O$), gallium chloride ($GaCl_3$), and selenium dioxide (SeO_2). The chemical compounds $AlCl_3.6H_2O$, $GaCl_3$, and SeO_2 contained 0.1 M, 0.1 M, and 0.01 M respectively in 400 milliliters of deionized water. The mixture was then stirred; the initial pH was measured and adjusted to 4.0. The ternary compound AlGaSe can be deposited between 1100 and 1700 mV. Figure 6 shows the cyclic voltammogram, which reveals a steady increase in current flowing through the electrolyte at about 1100 mV and a cathodic peak at about 1600 mV, with the maximum peak potential suggesting a suitable potential to deposit the semiconductor. Olusola et al. (2023) revealed that AlGaSe can grow at a cathodic voltage of 1200 mV; this voltage falls within the suitable voltage range obtained in this work for the growth of AlGaSe thin films [12].

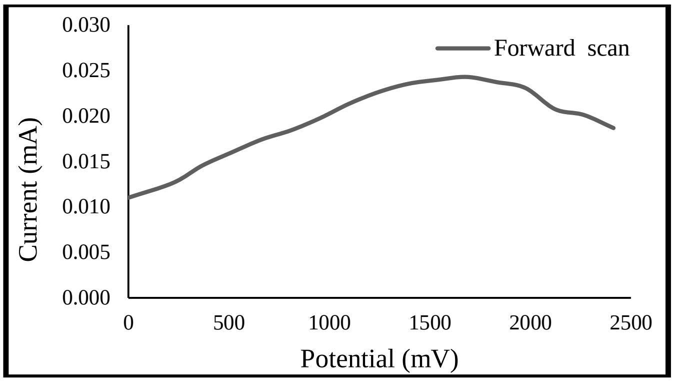

Figure 6: Cyclic voltammogram of aluminum gallium selenide (AlGaSe) using scan rate of 150 mV/s.

Table 1: Potential window obtained using the developed potentiostat compared to the potential range in literatures

Semiconductor Materials	Measured Cathodic Potential Window (mV)	Cathodic Potential Window in Literature (mV)
ZnS	1100 - 1600	1200 - 1650 [8]
CdS	900 -1500	900 - 1500 [10]
AlGaSe	1100 - 1700	1200 [12]

The findings from the analyses of zinc sulfide (ZnS), which yields a potential range of 1100–1600 mV in comparison to the findings of Madugu et al. (2016) [8], and cadmium sulfide (CdS), which yields a potential range of 900–1500 mV in accordance with the findings of Olusola (2016) [10], are summarized in Table 1. However, there is currently no literature detailing the suitable potential range to grow aluminum gallium selenide thin films. The work by Olusola et al. (2023) only revealed a single potential, and not range of potential to electrodeposit AlGaSe thin films [12]. It is necessary to use the available semiconductor characterization techniques, such as UV-visible spectrophotometry, scanning electron microscopy (SEM), X-ray diffraction (XRD), energy dispersive X-ray analysis (EDX), photoelectrochemical (PEC) cell, and DC conductivity measurements, to further determine the near stoichiometry of the compound semiconductors.

4. Conclusion

The development and validation of a low-cost and user-friendly cyclic voltammetry analyzer have been successfully achieved in this work. The developed instrument showed that ZnS has a cathodic potential range of 1100 to 1600 mV, which is in agreement with reports in the literature. The results indicate that the instrument performs well when compared to the range of cathodic potentials that have been established in the literature, with an average offset potential of ~75 mV for ZnS deposition. Material scientists and scholars can use the instrument for experimental research purposes to determine the appropriate potential range to deposit semiconductor materials in the laboratory. Future work will focus on expanding the tested material range and improving signal stability at lower scan rates. Overall, the results confirm that the developed analyzer provides a viable alternative for determining deposition parameters in semiconductor electrochemistry.

Acknowledgement
The Federal University of Technology Akure is acknowledged for providing the research facilities to execute this research.

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