DC measurement techniques for Hydrogen Evolution Reaction (HER)

Application Note: 2023/07

In this application note, we provide a brief overview of the standard DC measurement techniques and variable parameters that are to be considered when evaluating electro-catalysts for hydrogen evolution reaction (HER) using the *AMETEK Solartron Analytical – ModuLab XM* instrument, which is a versatile instrument for material electrochemistry and photovoltaic measurements.

Introduction

The hydrogen economy envisions hydrogen gas as a clean energy carrier, offering a solution to reduce emissions and transition from fossil fuels. Hydrogen's versatility makes it valuable across industries. One eco-friendly method to produce hydrogen is through water splitting, where water molecules are separated into hydrogen and oxygen gases. This can be achieved through water electrolysis, using renewable sources like solar or wind power, or through photolysis, which utilizes sunlight and specialized materials. Despite its potential, challenges remain. The reduction of costs in electrolysis technology is crucial, alongside the necessity to establish highly efficient infrastructure. Achieving this demands a significant investment in extensive research and development efforts.

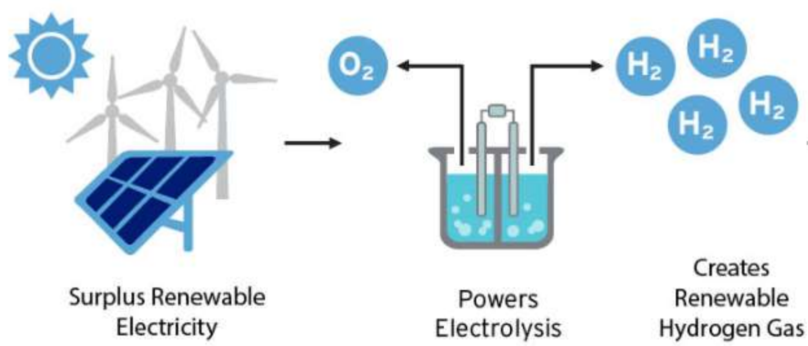

Figure 1. Schematic representation of renewable hydrogen generation through water-electrolysis

Electrochemical measurement techniques are vital in understanding and optimizing water-splitting reactions, which are essential for clean energy production and storage. This application note aims to provide guidance on utilizing Solartron Analytical instrument~~s~~ for three essential electrochemical techniques: Cyclic Voltammetry (CV), Linear Sweep Voltammetry (LSV), and Galvanostatic measurement. These techniques can yield valuable information about the kinetics, stability, and reaction mechanisms of water-splitting processes, particularly in the context of hydrogen evolution reactions.[0]

Instrumentation and Experimental Setup

For this application note, we will use Solartron Analytical ModuLab XM Instrument, which offers high precision and reliability in electrochemical measurements. The overall setup consists of a ModuLab XM (Potentiostat/Galvanostat, Frequency Response Analyzer), which has proven to be an outstanding electrochemical workstation for measurements of water-splitting reactions[2,3] and an electrochemical cell equipped with appropriate working, reference, and counter electrodes.

1. Electrochemical Cell Preparation: Use a three-electrode setup with a working electrode (usually a metal or metal oxide-based electrode), a reference electrode (e.g., Ag/AgCl), and a counter electrode (e.g., platinum or graphite). Ensure the cell is assembled correctly and free from air bubbles. Here we use Ni-foam as the working, Ag/AgCl as a reference, and a platinum wire as the counter electrode.

2. Electrolyte: Use a suitable supporting electrolyte, such as an aqueous solution of 1 M KOH or 1 M NaCl, to facilitate the water-splitting reactions.

3. Instrument Connection: Connect the working, reference, and counter electrodes to the corresponding probes on the PSTAT card of the Solartron Analytical – ModuLab XM instrument.

4. Software Setup: Launch the Solartron XM-Studio software and select the appropriate electrochemical technique (CV, LSV, or Galvanostatic) for measurements. Configure the experiment parameters (potential range, step size, duration, etc.) according to your specific requirements, or as suggested further in this application note. All the Potentials (V) have been converted within the software versus reversible hydrogen electrode (vs. RHE).

Techniques

1. Cyclic Voltammetry (CV)

Cyclic Voltammetry is a widely used technique to study redox processes and assess the electrochemical behavior of materials. It involves scanning the electrode potential linearly between two defined limits while measuring the resulting current, as shown in Figure 2. CV provides information about the reversibility of the redox reaction, peak potentials, and reaction kinetics.

Experiment Parameters:

- Potential range: Typically, from a negative value (e.g., -0.6 V vs. RHE) to a positive value (e.g., 1.2 V vs. RHE).
- Scan rate: Choose a suitable scan rate (e.g., 50 mV/s) for the desired time resolution.

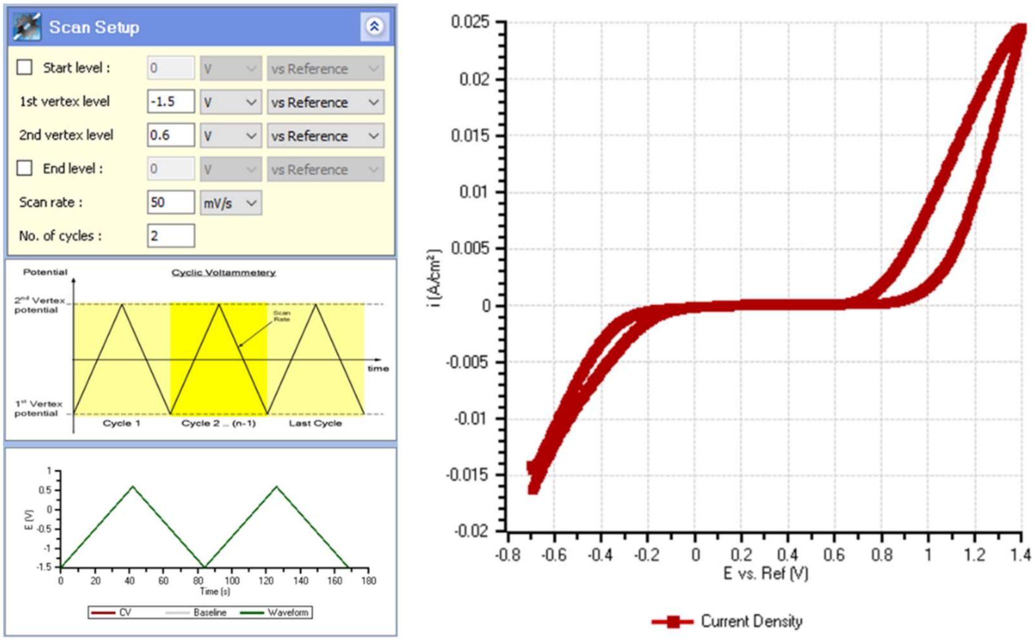

Figure 2. Cyclic Voltammetry (CV): variable parameters and measurement results.

2. Linear Sweep Voltammetry (LSV)

Linear Sweep Voltammetry is similar to CV but involves a continuous linear increase or decrease in the electrode potential. LSV, as shown in Figure 3, is used to determine the current response at different potentials and can help identify the onset/over the potential for hydrogen evolution reaction.

Experiment Parameters:

- Potential range: Set the starting and ending potentials based on your expected hydrogen evolution reaction region.
- Scan rate: Use a slow sweep rate to capture detailed information about the electrochemical response (e.g., 10 mV/s).

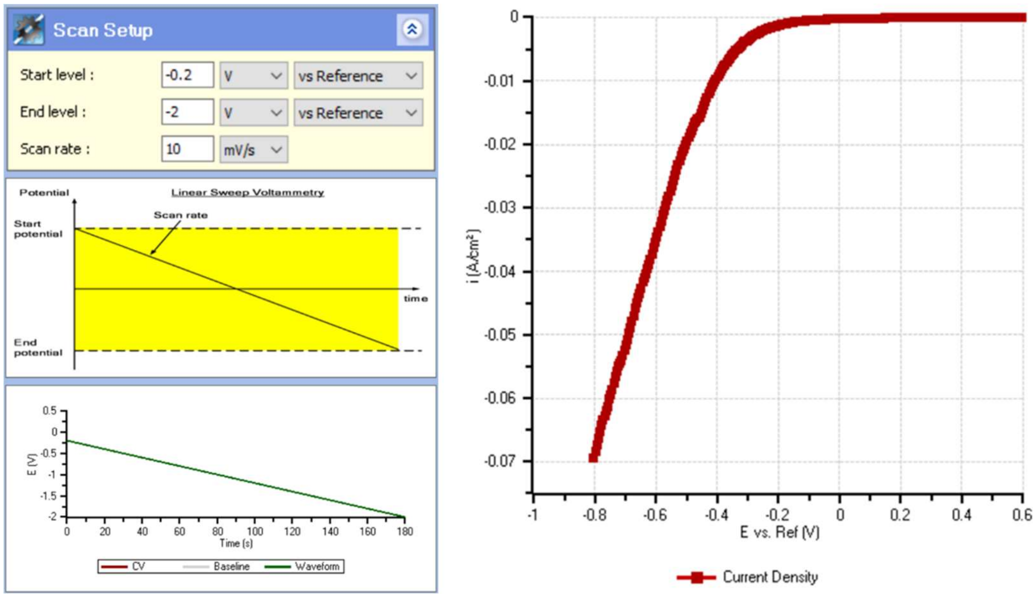

Figure 3. Linear scan voltammetry (LSV): variable parameters and measurement results.

3. Galvanostatic

Galvanostatic technique involves applying a constant current to the working electrode and monitoring the resulting potential or time-dependent current response. As shown in Figure 4, they are helpful in studying water-splitting kinetics under controlled conditions.

Experiment Parameters:

- Current level: Set the desired current density for the galvanostatic experiment.
- Duration: Choose an appropriate duration of the overall measurement.

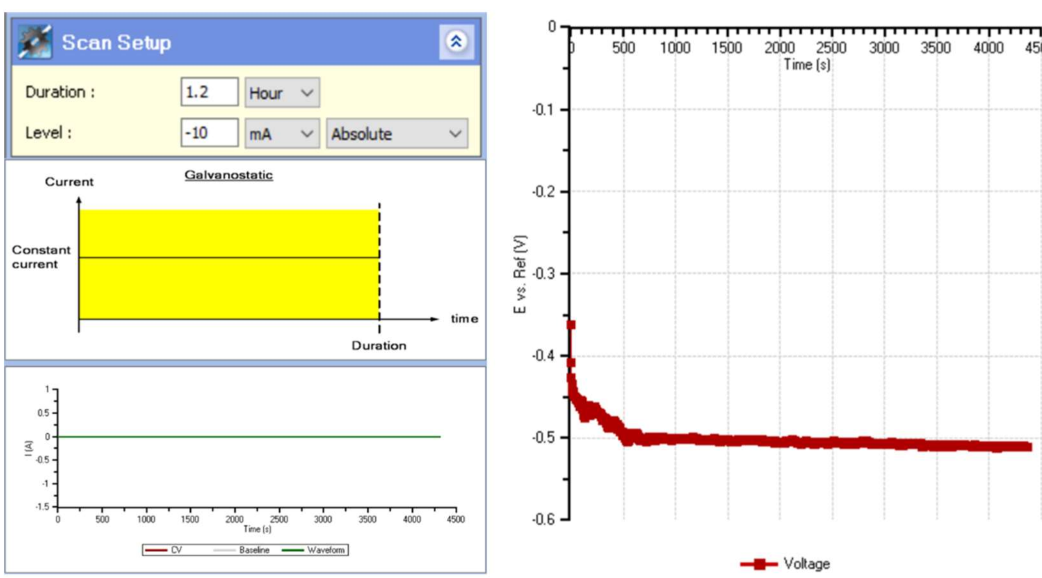

Figure 4. Galvanostatic: variable parameters and measurement results.

Data Analysis

After performing the experiments, the obtained data can be analyzed using the Solartron software or other data analysis tools. Herein, as an example, the Tafel slope for the hydrogen evolution reaction (HER) has been determined from the Linear sweep voltammetry (LSV) curve using the XM-Studio software,(Figure 5). For this, the curve has been plotted as the log of Current Density (mA/cm^2) as the x-axis and Voltage (mV) as the y-axis. Using the Line fitting method, the curve can be fitted for the region of interest (e.g., Log i (mA/cm^2) between 0 and 1) to determine the Tafel slope (gradient), which is found to be 220 mV/decade.

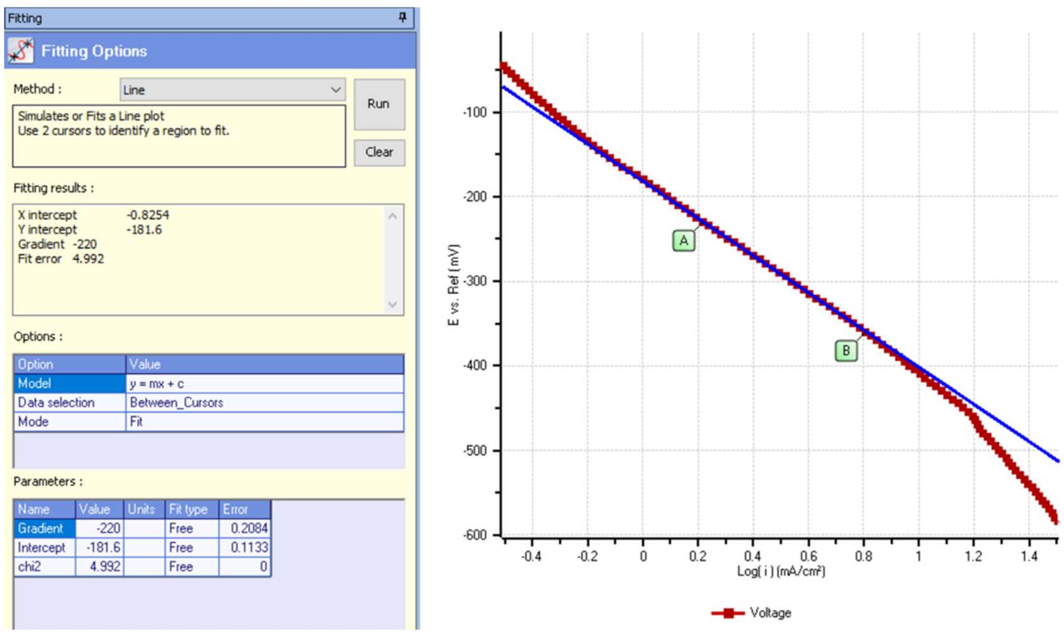

Figure 5. Line fitting of LSV curve for the determination of Tafel Slope.

Summary – In this application note, we have highlighted the electrochemical measurements for hydrogen evolution reactions (HER) of the water-splitting using Solartron analytical instruments. By employing Cyclic Voltammetry, Linear Sweep Voltammetry and Galvanostatic measurements, researchers can gain valuable insights into the mechanisms and kinetics of water-splitting, which is critical for advancing clean energy technologies. The combination of these techniques can lead to a comprehensive understanding of the water-splitting process and aid in the development of efficient and sustainable catalysts.

Please contact us for more information at **www.ameteksi.com**

References

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