Department of Materials Science and Engineering

Corrosion and Materials Protection Laboratory

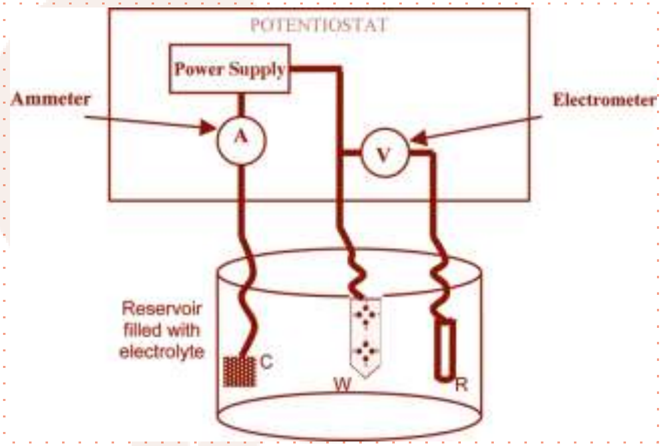
Analytical and non-analytical instruments

ANALYTICAL INSTRUMENTS

Potentiostat/Galvanostat

Introduction

In order for an electrochemical process to take place, there must be an anode, a cathode, as well as both an ionic and electrical conduction path between the two. When performing a DC polarization scan, as will be discussed in this application note, the ionic conduction path is provided through the solution separating the working and counter electrodes, while the electrical conduction path is provided through



Schematic presentation of a electrochemical test cell

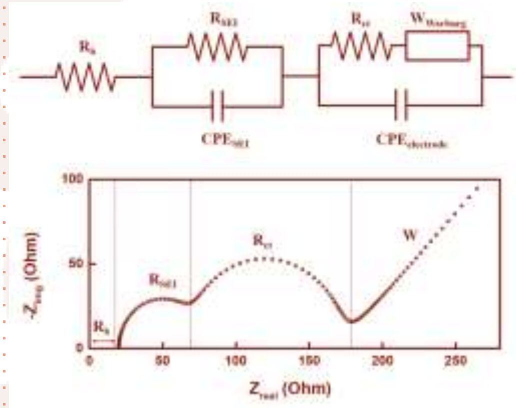
the **potentiostat**. This potentiostat is then used to control the driving force for electrochemical reactions taking place on the working electrode. The magnitude of this driving force in turn dictates which electrochemical processes actually take place at the anode and cathode, as well as their rate. The potential, is a thermodynamic property and provides information on whether or not a reaction can occur. The current, on the other hand, represents the rate with which the anodic or cathodic reactions are taking place on the working electrode. Typically, the current density is used rather than current itself.

Potentiostat/Galvanostat

Introduction

Electrochemical corrosion experiments measure and/or control the potential and current of the oxidation/reduction reactions. Several types of experiments are possible by manipulating and measuring these two variables.

Most experiments impose a potential on the working electrode and measure the resulting current, such as: potentiostatic and potentiodynamic experiments. Experiments where the current is imposed rather than the potential are referred to as **galvanodynamic** or galvanostatic.



Equivalent corrosion circuit and corresponding Nyquist diagram

Electrochemical impedance spectroscopy (EIS) is a powerful technique that utilizes a small amplitude, alternating current (AC) signal to probe the impedance characteristics of a cell. The AC signal is scanned over a wide range of frequencies to generate an impedance spectrum for the electrochemical cell under test. EIS differs from direct current (DC) techniques in that it allows the study of capacitive, inductive, and diffusion processes taking place in the electrochemical cell.

Potentiostat/Galvanostat

Introduction

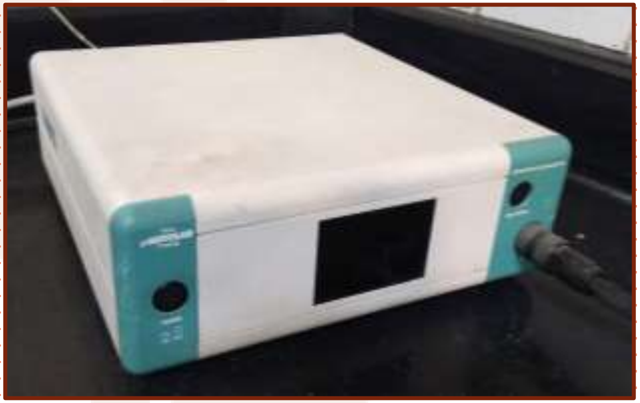
As mentioned earlier, potentiostats are used to keep the potential (voltage) between a working electrode and a reference electrode at a constant value. Galvanostats are used to maintain a constant flow of current through an electrolytic cell.

Potentiostats and galvanostats are electrochemical instruments used in electrochemistry, battery and fuel cell testing, corrosion control, voltammetry, biomedical research, surface imaging, and related applications.

Device: Potentiostat/Galvanostat

Manufacturer: *METROHM AG*

This lab is equipped with two potentiostat/galvanostat devices which the technical specification for each will be presented.



Technical specification

Electrode connections	**2, 3**
Potential range	**± 5 V**
Compliance voltage	**± 12 V**
Maximum current	**± 80 mA**
Current ranges	**10 mA to 10 nA**
Potential accuracy	**± 0.2 %**
Potential resolution	**3 µV**
Current accuracy	**± 0.2 %**
Current resolution	**0.0003 % (of current range)**
Input impedance	**> 100 GOhm**
Potentiostat bandwidth	**500 kHz**
Computer interface	**USB**
Control software	**NOVA**
impedance measurements frequency	**10 µHz - 500 kHz**

Device: Potentiostat/Galvanostat

Manufacturer: *IVIUM TECHNOLOGIES*

Technical specification

Current compliance	**±5A**
Maximum compliance voltage	**±10V**
Maximum applied voltage	**±10V below 1A, and ±8V up to 5A**
Electrode connections	**4; WE, CE, RE, S (and GND) with 4mm banana plugs**
Potentiostat bandwidth	**8 MHz**
Stability settings	**High Speed, Standard and High Stability**
Programmable response filter	**1 MHz, 100 kHz, 10 kHz, 1 kHz, 10 Hz**
Signal acquisition	**Dual channel 24bit ADC, 100,000 samples/s**
Applied potential range	**±10 V, 0.02 mV res. (20bit)**
Applied potential accuracy	**0.2% or 1 mV**
Current ranges	**±1 pA to ±10 A**

Device: Potentiostat/Galvanostat

Manufacturer: *IVIUM TECHNOLOGIES*

Technical specification (cont.)

Measured current resolution	**0.00001% of current range, minimum 0.6 aA**
Measured current accuracy	**10 pA + 0.025% of FSR**
Applied current resolution	**0.00013% of applied current range**
Applied current accuracy	**0.2%**
Galvanostatic current ranges	**±100 pA to ±10 A**
Potential ranges	**±1 mV, ±4 mV, ±10 mV, ±40 mV, ±100 mV, ±400 mV, ±1 V, ±4 V, ±10 V**
Measured potential resolution	**0.00001% of potential range, minimum 0.15 nV**
Measured potential accuracy	**0.2% or 1 mV**
Frequency range	**10 µHz to 8 MHz**
Amplitude	**0.015 mV to 1.0 V, or 0.03% to 100% of current range**

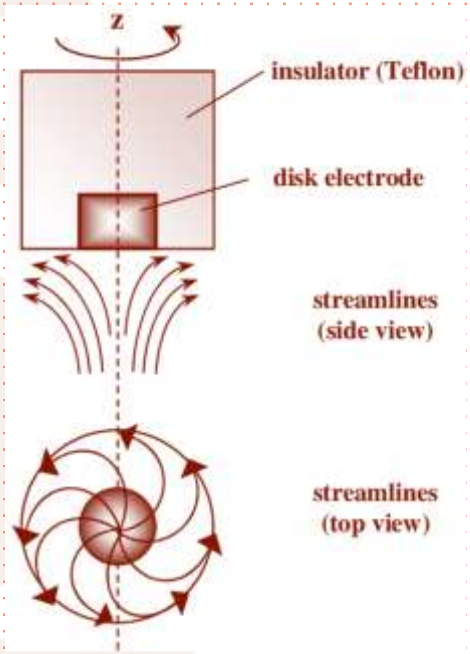
Technical specification (cont.)

Input impedance	**>1000Gohm //<8pF**
Input bias current	**<10pA**
Bandwidth	**>16MHz**
Ohmic drop compensation	**2V/current range, 16bit resolution**
Dynamic range	**0.05nV to 10V, and 0.2aA to 5A**

Rotating disc electrode

Introduction

The rotating disc electrode (RDE) is the classical hydrodynamic electroanalytical technique used to limit the diffusion layer thickness. Forced convection has several advantages which include: 1) the rapid establishment of a high rate of steady-state mass transport and 2) easily and reproducibly controlled convection over a wide range of mass transfer coefficients. The RDE consists of a disc (e.g. of Pt, Ni, Cu, Au, Fe, Si, CdS, GaAs, glassy carbon and graphite) set into an insulating (PTFE) surround. The electrode is rotated about its vertical axis, typically between 400 and 10,000 rpm. The theory for the hydrodynamics at the RDE assumes that the electrode is uniformly accessible



Schematic representation of streamlines during RDE experiment

and affords a precise and reproducible control of the convection and diffusion of reactant to the electrode. Hence, the RDE can be used to study the kinetics of interfacial processes. This lab is equipped with a RDE which its specifications are introduced.

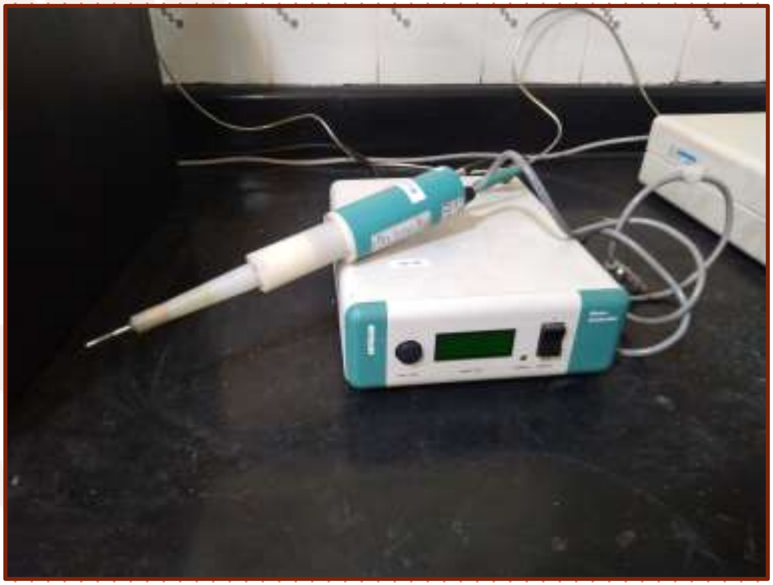
Device: Rotating disc electrode

Manufacturer: *METROHM AG*

This lab is equipped with a rotating disc electrode device with the following specifications.

Technical specification (cont.)

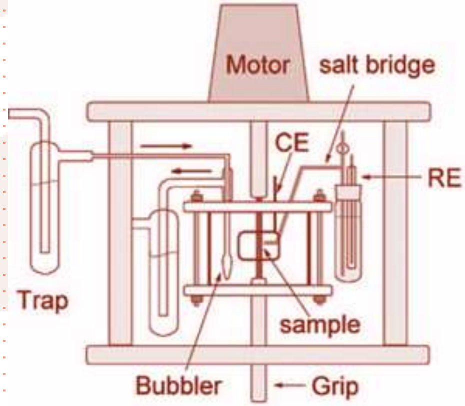
Rotation rate	**100-10000 rpm**
Rotation rate precision	**1 rpm**
Acceleration and deceleration rate	**4000 rpm/s**



Slow strain rate testing

Introduction

Slow strain rate testing (SSRT)), also called constant extension rate tensile testing (CERT), is a popular test used by research scientists to study stress corrosion cracking. It involves a slow (compared to conventional tensile tests) dynamic strain applied at a constant extension rate in the environment of interest. These test results are compared to those for similar tests in a, known to be inert, environment. The evaluated parameters are:



Schematic representation of SSRT test where the specimen is held in a corrosion test cell

- time to specimen failure (e.g., breakage, or from other "failure" criteria)
- ductility (by elongation to fracture or the reduction of the area)
- ultimate tensile strength (from the maximum load)
- area under the elongation - load curve (which represents the fracture energy)
- percent of ductile/brittle fracture on the fracture surface
- threshold stress for cracking

Slow strain rate testing

Introduction

The results of the SSRT tests are evaluated using the ratio:

$$\frac{\boldsymbol{result\ from\ specimen\ in\ test\ enviroment}}{\boldsymbol{result\ from\ specimen\ in\ inert\ enviroment}}$$

The departure of the ratio below unity quantifies the increased susceptibility to cracking. The test is best used in combination with electrochemical measurements and other stress corrosion cracking tests.

Device: Slow strain rate testing machine

Manufacturer: *ROYIN GRAN SANAT GHARB ASIA*

This lab is equipped with a SSRT with the following technical specifications:

Technical specification

Maximum load	**5 ton**
Load precision	**1 kg**
Strain rate	**10-7-10-4 (1/s)**
Strain rate precision	**1%**
Computer interface	**USB**
Output format	**txt, xlsx, html**

- **Continues monitoring of load and displacement and plotting force-displacement curve**