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Some Practical Ideas for the Establishment of an Electrochemical Teaching Laboratory*

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This paper describes one approach to the problems of establishing a basic corrosion and electrochemistry teaching laboratory in a developing university with tight financial constraints. The initial success of the project suggests that it may be of interest to other institutes with similar goals and difficulties.

INTRODUCTION

ANYONE who has considered setting up a teaching laboratory in recent years will be aware of two factors:

1. Because of the relatively high expense of running practical courses, they are not currently being expanded as a method of teaching undergraduates in many countries, despite the benefits of laboratory experience to the students and the industries that ultimately employ them.
2. The capital cost of specialist laboratory equipment is high and limited demand, due to financial constraints within institutions, prevents savings being achieved through large batch production methods.

In this paper we attempt to address some of the problems of providing equipment for effective teaching through practical classes while working to a limited budget. We describe the production of economic and robust potentiostat and galvanostat units and their use to demonstrate the principles of electrochemistry as part of the development of an undergraduate corrosion laboratory at the University of Zambia (UNZA).

Like most universities in developing countries, UNZA is a state university and relies entirely on government funding for all of its activities. One of the problems identified when a corrosion course was introduced at UNZA was how an associated laboratory programme was going to be conducted to complement the lectures, considering the lack of resources in the Department of Metallurgy and Mineral Processing and the limited availability of funds. In addition, since the manufacturing industry in Zambia is still in its infancy, almost all commercial equipment required to set up a corrosion laboratory is expensive and can generally only be imported from other countries outside of Africa. It was decided to collect information on an existing laboratory course and to see if the ideas gathered could be adapted to enable a course to be prepared at UNZA, without incurring great expense or taxing the resources of the Department too heavily.

The Department of Metallurgy and Mineral Processing at UNZA has a British Council link with the Materials Division at the University of Newcastle upon Tyne. Assistance was sought to provide funds for a short visit to UNZA by a corrosion scientist during 1994 and for some low-cost components to be supplied for this project.

Over the years, since the development of the transistor and microchip, electronics has advanced rapidly. The diversity and performance of equipment available today for electrochemical measurement is extensive. However, the problems of cost and the need for simple, reliable systems for teaching and research laboratories have long been recognized [1]. The potentiostat is perhaps the most fundamental electronic device for performing corrosion and electrochemical studies today. During an electrochemical reaction there will be changes in the reaction cell due to alterations in the surface condition of the electrodes, local solution changes, cell resistance changes etc. In order for a reaction to be studied, it is necessary to decrease the number of variables in the system. The potentiostat is thus used to keep the potential of the electrode under study constant while allowing the reaction current to be monitored. The

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magnitude, direction and changes in the current flow provide information about the kinetics and equilibration of the cell. Similarly, if the current flowing through the electrode is kept constant (galvanostatic control), the potential may be monitored and the data used to provide thermodynamic information about the electrode reactions. Constant current measurements also yield quantitative data about reactions.

The potentiostat's function is to maintain a set potential between a test specimen and a reference potential source by driving an appropriate current through the specimen. The potential is maintained, regardless of changes occurring on the specimen surface or in the electrolyte, by adjusting the current flowing from the power supply between the specimen and an inert counter electrode (usually platinum, but stainless steel will do for many applications), and using a feedback loop employing the electrode potential constantly monitored from the cell. Several authors have published designs for potentiostats [2]. The rapidly advancing nature of the electronics field and the particular requirements for teaching laboratory equipment have led us to develop alternative circuits for the laboratory experiments, which are described later. An add-on unit was constructed so that current-controlled experiments could be performed and this is also described. The components for these units were partially selected on the basis of their low cost and ease of procurement from electronic component companies.

Circuit details

The potentiostat and galvanostat circuits were designed at Newcastle.

The potentiostat unit is based on an LM759CP power op-amp and the circuit details are shown in Figs 1 and 2. Figure 1 shows the mains power supply built to drive the unit which consists of a 20 VA, 12-0-12 V rms, transformer, a rectifier block and two smoothing capacitors. Alternatively, two 12 V batteries could be used to provide the split rail supply for the potentiostat, resulting in a small decrease in the compliance voltage of the unit. The potentiostat circuit is shown in Fig. 2, and pin connections for the 2.5 V precision voltage reference diodes (left) and the integrated circuit (right) are also shown. The unit is capable of generating ±2.5 V at a maximum current of 0.5 A. Switch 'A' enables the polarization of the specimen to be moved through zero from anodic to cathodic while the magnitude of the potential is adjusted using the ten turn potentiometer labelled 'B' in Fig. 2. Switch 'C' allows the electrodes to be isolated from the unit. The circuit does not require any calibration once constructed. External meters can be used to measure the potential and current flow. The behaviour of the unit may, however, be checked by connection to a 'dummy cell' made up of two 1000 Ω resistors in series. The reference electrode wire is linked to the centre and the working and counter electrode connections to the two ends of the 'dummy cell'. An ammeter is placed in the counter electrode line and a voltmeter is placed across the working and reference electrode. Measurements of voltage and current are recorded and then plotted out. The graph obtained should be linear with a slope equivalent to the value of the resistor between the reference and working connections.

Figure 3 shows the circuit diagram for the add-on galvanostat unit, this device provides a constant current supply whose magnitude may be adjusted

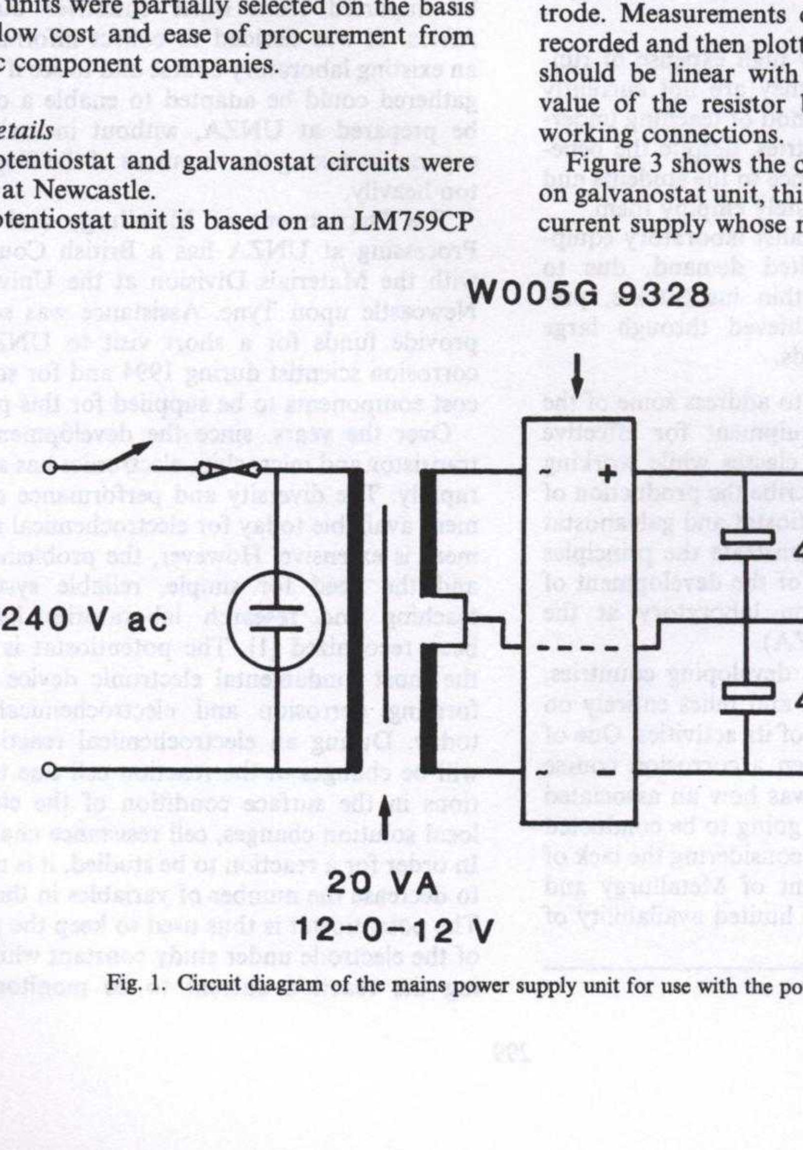

Fig. 1. Circuit diagram of the mains power supply unit for use with the potentiostat.

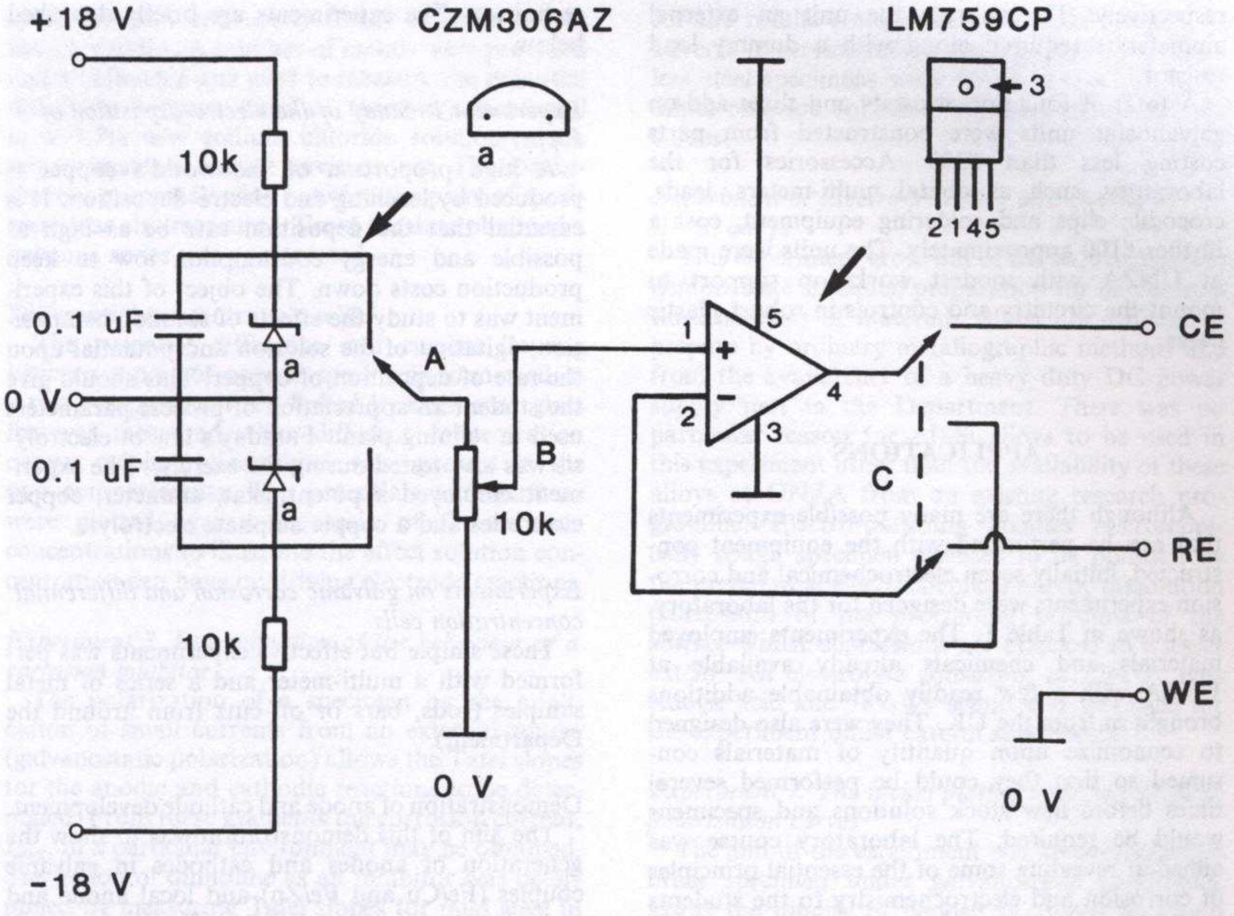

Fig. 2. Circuit diagram for the teaching laboratory potentiostat showing the device pin connections.

using the potentiometers marked 'Coarse' and 'Fine' in the figure and the potentiometer, B, on the potentiostat. The current may be read from the $\pm 25\,\mu$A centre zero meter by multiplying the reading by the scaling factor on the wafer switch (three-pole, three-way type). The unit will thus provide galvanostatic outputs of $\pm 25\,\mu$A, $\pm 250\,\mu$A and ± 25 mA (the direction of current flow of the output being controlled by switch 'A' on the potentiostat). This galvanostat current meter has the ×10 and ×1000 ranges calibrated by adjustment of the 100 and 10 Ω variable resistors

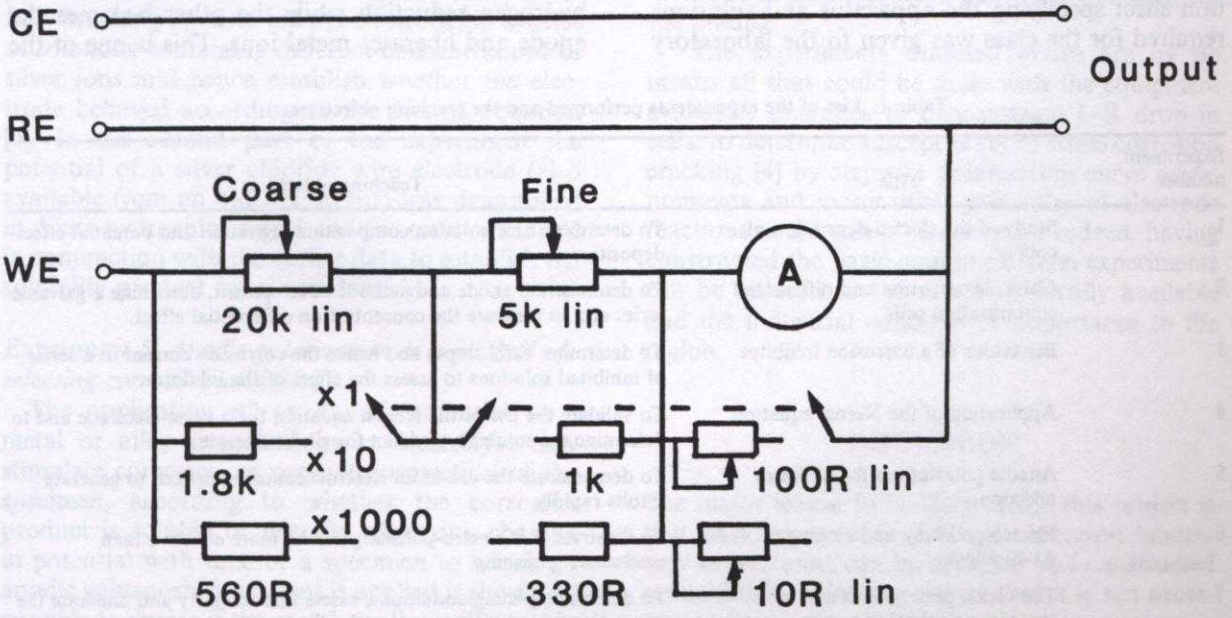

Fig. 3. Circuit diagram for the add-on galvanostat unit employing a 25 μA moving coil ammeter.

respectively. To calibrate the unit an external ammeter is required along with a dummy load resistor.

A total of four potentiostats and three add-on galvanostat units were constructed from parts costing less than £300. Accessories for the laboratory, such as digital multi-meters, leads, crocodile clips and soldering equipment, cost a further £100 approximately. The units were made at UNZA with modest workshop support to mount the circuitry and controls in robust plastic cases.

APPLICATIONS

Although there are many possible experiments that can be performed with the equipment constructed, initially seven electrochemical and corrosion experiments were designed for the laboratory, as shown in Table 1. The experiments employed materials and chemicals already available at UNZA with a few readily obtainable additions brought in from the UK. They were also designed to economize upon quantity of materials consumed so that they could be performed several times before new stock solutions and specimens would be required. The laboratory course was aimed at revealing some of the essential principles of corrosion and electrochemistry to the students by studying systems of which some were related to the local industries. An instruction sheet was prepared for use by the students for each experiment and this included an introduction, references for further study, details of how the data should be treated and some questions to answer. The students were intended to work in groups of two or three so that they could divide the labour between themselves and assist each other in interpretation of the results. A second instruction sheet specifying the apparatus and solutions required for the class was given to the laboratory technician. The experiments are briefly described below.

Experiment 1. Study of the electro-deposition of copper

A high proportion of the world's copper is produced by leaching and electro-deposition. It is essential that the deposition rate be as high as possible and energy consumption low to keep production costs down. The object of this experiment was to study the effects of solution composition, agitation of the solution and potential upon the rate of deposition of copper. This should give the student an appreciation of process parameters used in refining plant. Faraday's law of electrolysis was also tested during the exercise. The experiment employed a potentiostat, ammeter, copper electrodes and a copper sulphate electrolyte.

Experiments on galvanic corrosion and differential concentration cells

These simple but effective experiments was performed with a multi-meter and a series of metal samples (rods, bars or off-cuts from around the Department).

Demonstration of anode and cathode development. The aim of this demonstration was to show the generation of anodes and cathodes in galvanic couples (Fe/Cu and Fe/Zn) and local anode and cathode areas on single metal surfaces using specimens suspended in a chloride containing agar gel with a pH (phenolphthalein) and an iron species indicator (potassium hexacyanoferrate III) present to indicate areas of alkalinity and iron dissolution around the specimens.

The galvanic series. If dissimilar metals are coupled one electrode becomes the cathode and supports oxygen or hydrogen reduction while the other becomes the anode and liberates metal ions. This is one of the

Table 1. List of the experiments performed and the teaching objectives

Experiment number	Title	Teaching objectives
1	Study of the electro-deposition of copper	To determine how solution composition, agitation and potential effect deposition.
2	Galvanic corrosion and differential concentration cells	To demonstrate anode and cathode development, determine a galvanic series and to measure the concentration differential effect.
3	Behaviour of a corrosion inhibitor	To determine Tafel slopes and hence the corrosion current in a series of inhibited solutions to assess the effect of the inhibitor.
4	Application of the Nernst equation	To validate the use of the Nernst equation for a silver electrode and to determine the solubility product for silver chloride.
5	Anodic polarisation for inhibitor selection	To demonstrate the use of an electrochemical approach to generate results rapidly.
6	Electro-polishing and etching of Al–Si alloys	To illustrate that electro-polishing can be more effective than mechanical polishing.
7	The nickel plating bath and stepwise polarisation curves	To determine plating conditions, assess film integrity and illustrate the generation of polarisation curves by the stepwise method.

most common and costly forms of corrosion in industry today. A number of metals were provided and a voltmeter was used to measure the potential difference between pairs of specimens immersed in a 3.5% w/w sodium chloride solution which approximates a marine environment. The instantaneous current flowing between the pairs of specimens was also measured. From the data obtained a galvanic series was constructed.

Differential concentration cells.

The potential difference and current flowing between a pair of copper specimens immersed in a two-compartment cell linked by a sintered glass frit was measured when different concentration copper sulphate electrolytes were present in the two compartments. The potentials and currents were plotted against the ratio of the solution concentrations to illustrate the effect solution concentration can have on driving electrode reactions.

Experiment 3. Determination of the behaviour of a corrosion inhibitor.

The polarization of a specimen by the application of small currents from an external source (galvanostatic polarization) allows the Tafel slopes for the anodic and cathodic reactions to be determined. From these gradients the corrosion current, I_{corr}, in a particular environment may be obtained. The action of quinoline as an inhibitor was determined by measuring Tafel slopes for mild steel in sulphuric acid with 0, 0.1, 1 and 2% v/v quinoline present. An acid and inhibitor environment is often used for pickling steel. The inhibitor was tested to see if it acted by adsorption on the steel by use of Langmuir theory [3].

Experiment 4. Test of the application of the Nernst equation

The aim of this experiment was to measure the potential of a silver electrode (obtained by carefully breaking open an old pH electrode) in a series of solutions containing different concentrations of silver ions and hence establish whether the electrode behaved according to the Nernst equation [3]. In the second part of the experiment the potential of a silver chloride wire electrode (also available from an old pH sensor) was determined in dilute hydrochloric acid and the result was used in conjunction with the earlier data to establish the solubility product for silver chloride.

Experiment 5. Anodic polarization as a method of selecting corrosion inhibitors

The application of an anodic potential to a metal or alloy immersed in an electrolyte may stimulate corrosion or promote protection of the specimen, according to whether the corrosion product is soluble or not. By observing changes in potential with time of a specimen to which an anodic galvanostatic current is applied it should be possible to decide if the metal or alloy will corrode or not in the environment, without having to wait for the results from prolonged exposure tests or adverse in-service failure reports. Mild and stainless steel specimens were tested in tap water and dilute chloride solutions adjusted to various pH values.

Experiment 6. Electro-polishing and etching of Al–Si alloys

This experiment arose out of the requirements to demonstrate specimen preparation for microstructural analysis of materials which are difficult to prepare by ordinary metallographic methods and from the availability of a heavy duty DC power supply unit in the Department. There was no particular reason for Al–Si alloys to be used in this experiment other than the availability of these alloys at UNZA from an existing research programme. Electro-polishing enables microscopically rough specimen surfaces to be polished by the preferential electrochemical anodic dissolution (corrosion) of the protruding portions of the surface whilst depressions are attacked to a lesser extent. An electrolyte consisting of 22% v/v perchloric acid and 78% v/v acetic acid was used for the experiment under careful supervision.

Experiment 7. The nickel plating bath and stepwise polarization curves

The aim of the experiment was to electroplate a brass specimen under galvanostatic conditions, assess the quality of deposit produced by visual examination and determine its corrosion resistance in comparison with unplated brass by making stepwise polarization curve measurements with a potentiostat unit. The experiment indicates the importance of correct plating conditions in the production of protective and decorative coatings while also demonstrating a method of manual acquisition of polarization curves. A traditional 10% w/w nickel sulphate bath containing boric acid and sodium chloride additions was used for the plating.

The experiments outlined above are by no means all that could be done with the equipment available. Exercises to demonstrate I–R drop in cells, to determine susceptibility to stress corrosion cracking [4] by stepwise polarization curve measurements and many other principles of electrode reactions could easily be designed. Indeed, having constructed the basic equipment then experiments can be tailored to the materials locally available and the industrial concerns of importance to the region.

DISCUSSION

The major lesson to be learnt from this project is that basic electrochemical and corrosion laboratory experiments can be designed and constructed economically. We have shown that it is not necessary to have expensive, imported equipment to run an effective corrosion laboratory. Results can be achieved with relatively simple, robust designs which minimize the need for maintenance and costly repairs by outside agencies. This is particularly important for universities in developing countries such as Zambia, where the availability of financial and technical resources are limited. We estimate that in terms of consumables each experiment costs less than £2 to run using laboratory grade chemicals and re-using the electrodes. Thus the whole class would use around £10 of materials and as the students usually work in groups of two or three then the cost per head is far less. This cost is small compared with the benefits derived from 'hands-on' learning which enable the student to take what they often consider to be abstract theory and put it into practice. Indeed, the initial response from students who have had an opportunity to do the practicals is extremely positive and further development of practical classes is planned in related subject areas.

Students of UNZA can now conduct experiments to learn the principles of electrochemistry and corrosion. They can use information gathered in lectures to tackle practical problems and thus become aware of the importance of the field to their country's developing industry. The laboratory approach to teaching is probably the most effective available in that it actively engages the students in study while also allowing them to develop their practical skills. Although effort and some expenditure is required to teach in this way, we believe it should form an essential part of any undergraduate's training in engineering. Indeed, the experience acquired in practical classes will place new graduates in a better position to appreciate and operate in the industrial environment.

CONCLUSIONS

1. We have established an effective teaching approach to corrosion studies at UNZA by complementing the lecture programme with appropriate laboratory work.
2. We have demonstrated that laboratory classes may be developed economically utilizing available equipment and supplementing this with some basic electronics.
3. We have also shown that experiments can be designed to maximize student benefit while keeping running costs low.

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