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A high output power (70 V, 1.5 A) potentiostat-galvanostat

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The design of a high-power potentiostat–galvanostat is presented. This instrument is capable of driving a cell within the rectangular region 0 to ± 72 V and 0 to ± 1.5 A. The anodic oxidation of titanium in acidic medium is given as an example of its application.

1. Introduction

It is generally easy to devise suitable regulating devices for any type of electrochemical interface (metal or semi-conductor/electrolyte). The technological evolution of integrated circuits during recent years has allowed the improvement of the performance of these devices especially with respect to response time, bandwidth and stability. However the range of voltages which can be applied to the working electrode is generally limited to a few volts and the available power to a few tens of watts using such readily made devices.

The study of the anodic oxidation of some metals, electrochemical polishing or high resistivity organic electrolytes, need polarization voltages as high as some tens of volts. In addition, for example in the case of valve metals (aluminium, tantalum, titanium, etc.), during potentiostatic oxidation, a very high transient current is observed related to the oxide layer formation. Hence it is necessary to devise a potentiostat which does not saturate at high currents.

A high power regulating device has been built (± 70 V, 1.5 A) suitable for impedance measurements over a wide frequency range. This device was designed for both a potentiostatic and galvanostatic mode of control.

2. Principles of the device

2.1. *Potentiostat*

The usual potentiostatic arrangement using operational amplifiers [1] are recalled in Table I. There the transfer function is given for each circuit assuming that the operational amplifiers output impedance is negligible ($R_s \simeq 0$) and input impedances are very high ($Z_d \to \infty$). No voltage follower is needed in potentiostat A [2] since the input impedance seen by the reference electrode is sufficiently high and of the same order of magnitude as the input impedance seen from the source, E, i.e.

$$Z_e = A_1 Z_d \frac{Z_2}{Z_1 + Z_2}.$$

Even if this configuration is comparatively trouble-free (in particular, stability is easily achieved largely due to the use of only one operational amplifier in the feedback loop), it is often necessary to use circuit B because the high performance operational amplifiers A_1, as the bipolar operational power supply used, are often of the inverting type only. Voltage follower, A_2, in circuit B enhances the input impedance seen by the reference electrode. The circuit is arranged as a conventional inverting amplifier and the gain is given by the ratio of the impedances found at the input. This circuit is very useful when high voltage regulation is required.

2.2. *Galvanostat*

Control of the current flowing through electrochemical cells is effected by galvanostats (i.e., voltage to current transducers [3]). Simple configurations assume a 'floating' cell but if the working electrode is necessarily connected to earth, the two circuits C and D of the Table I can be used.

The simpler galvanostat (C) controls a current

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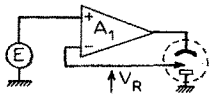
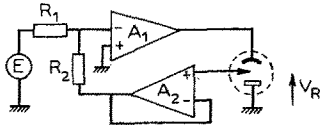
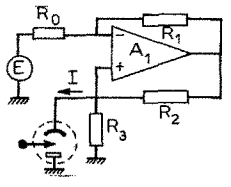
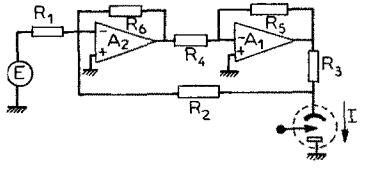
Table 1. Potentiostat and galvanostat circuits and definitions. Same definitions as in Fig. 1 with $Z = Z_1 + Z_2$

	Circuit	*Transfer function* V/E *or* I/E	*Conditions*
A		$\dfrac{1}{1 + (1/A_1)[(Z_1 + Z_2)/Z_2]}$	if $A_1 \to \infty$ $V_R = E$
B		$\dfrac{-(R_2/R_1)}{1 + (1/A_1)[(Z_1 + Z_2)/Z_2]\,[1 + (R_2/R_1)]}$ $A_2 \to \infty$	$V_R = -E(R_2/R_1)$
C		$\dfrac{-1}{(R_0/R_1)\{R_2[1 + (Z/R_3)]\} - Z + (1/A_1)[1 + (R_0/R_1)]\,(R_2 + (R_2Z/R_3) + Z)}$ if $(R_0/R_1) = R_3/R_2$ $\dfrac{-1/R_3}{1 + (1/A_1)[1 + (R_1/R_0)]\{1 + (Z/R_3)[1 + (R_0/R_1)]\}}$	$I = -E/R_3$
D		$\dfrac{(R_6R_5)/(R_1R_4)}{R_3 + Z[1 + (R_3/R_2) - (R_6R_5)/(R_2R_4)] + [(R_4 + R_5)/(A_1R_4)]\{R_3 + Z[1 + (R_3/R_2)]\}}$ if $1 + (R_3/R_2) = (R_6R_5)/(R_2R_4)$ and $R_1 = R_4 = R_5 = R_6$: $\dfrac{1}{R_3 + [(R_4R_5)/(A_1R_4)]\{R_3 + Z[1 + (R_3/R_2)]\}}$ $A_2 \to \infty$	$I = E/R_3$

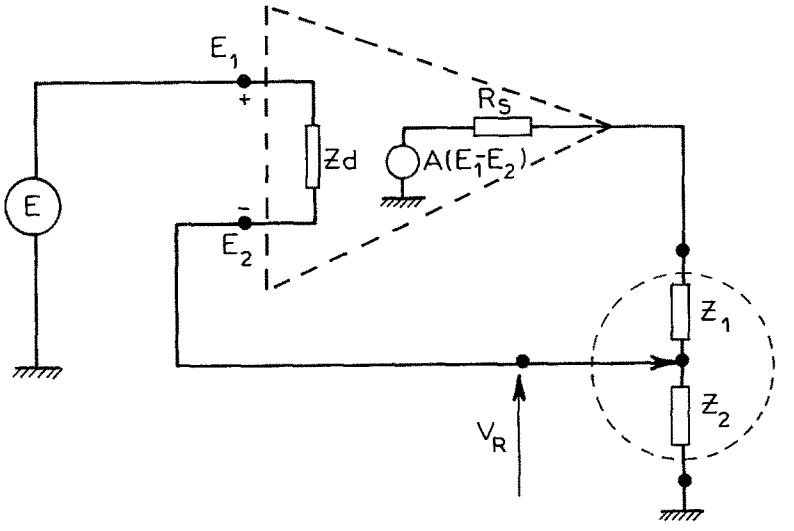


Fig. 1. Equivalent circuit of an operational amplifier with: E voltage source; V_R Reference electrode voltage; E_1, E_2 input voltage; Z_1, Z_2 equivalent circuit of the electrochemical cell; Z_1, impedance between counter electrode and reference electrode; Z_2, impedance between reference electrode and working electrode; A, open loop gain; Z_d, differential input impedance; R_s, output resistance.

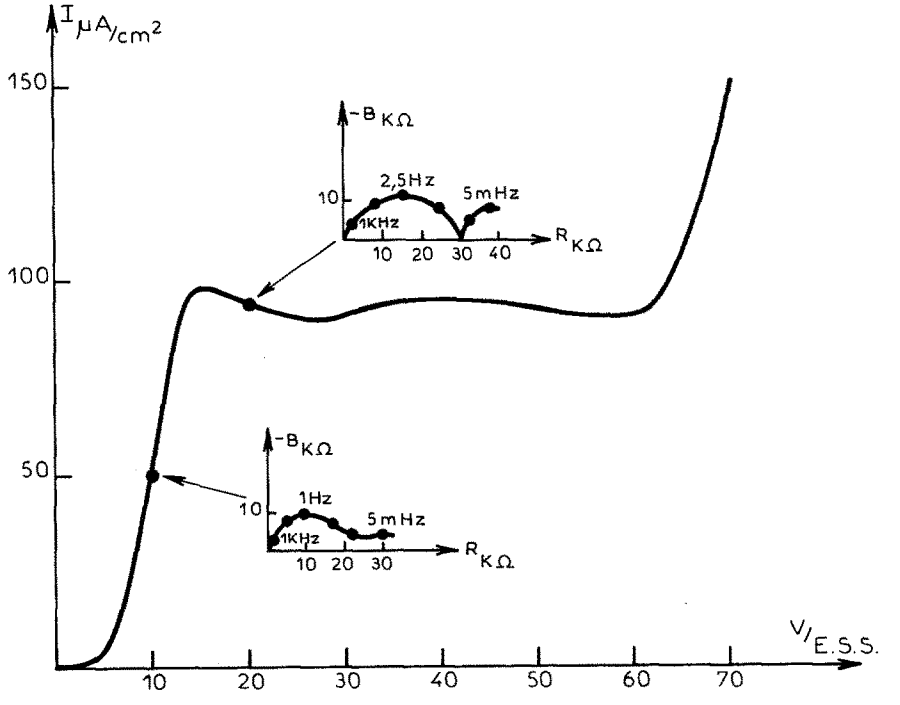


Fig. 2. Steady-state current–potential curve of Ti in 1 N H_2SO_4, with faradaic impedances diagrams [10].

of the opposite sense to that of the source and, therefore, can be redeployed as a negative output impedance regulator of especial interest in the study of multi-steady state systems [4].

Configuration D is well-suited to the design of galvanostats of high output power since the bipolar operational power supply used as A_1 requires that the non-inverting input should be grounded [5].

It is important to verify that the resistances in both circuits C and D should conform precisely with the selected relationships if the simple transfer functions given in Table I are to be obtained and if good control of the current is to be achieved.

3. Applications

The power amplifier chosen for the high power potentiostat–galvanostat was the bipolar operational power supply KEPCO BOP 72–1.5 M and the high voltage op-amp Burr-Brown BB 3582J for the voltage follower A_2. The main characteristics of these units are listed in Table 2.

Circuit B of Table 1 realizes a potentiostat with a 3 dB bandwidth of 17 kHz (by changing the value of the internal feedback capacitor of the bipolar operational power supply a 65 kHz bandwidth is easily obtainable at the expense of stability).

A time-delay circuit was incorporated for switching over from potentiostatic to galvanostatic modes. Diode input protection was also included.

This instrument has been used to study the anodic oxidation of titanium over a very large voltage range. The current–potential curve of a titanium electrode (UT 40, from Ugine Kuhlmann) in 1 N H_2SO_4 is given in Fig. 2. The anodic part only has been plotted since the cathodic part is well known [6, 7]. The steady-state polarization point is not easy to obtain because a relatively long time is needed to establish this condition whilst after 15 min the current will increase due to an intergranular corrosion.

Finally, this potentiostat has made possible the study of the optical and crystallographic properties [8] of the oxide layer. The growth law (2.5 nm V^{-1}) and the nature (anastase-microcrystalline form up to 50 V and crystallized form above 50 V) of the oxide layer have been established [9]. In addition, thanks to this device valence levels of the titanium oxide have been studied by ESCA in the Institut de Physique Nucléaire in Lyon.

Table 2. Amplifier specifications

	BOP 72-1.5M	BB 3582J
output voltage	± 72 V	± 145 V
output current	± 1.5 A	± 25 mA
unity gain bandwidth (small signal)	20 kHz	3 MHz
open loop gain	90 dB	118 dB

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