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Information

Application of Potentiostats Using PC in the Phase Analysis of Galvano-Chemistry

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Abstract: A set of device is designed based on PC to realize the functions of traditional potentiostats in the phase analysis of galvanochemistry. In the device, A/D and D/A conversion is used to measure the electrode potential and convert the voltage for output; specific program runs in the PC to regulate and control the system. The effect of the proportion factor K_P on the control variation is researched through certain experiment. Two experiments, the control of electrolysis in constant potential and the determination of polarization curves, are designed to test and evaluate the effectiveness of the device. The experimental results show that the variation between the actual anode potential and the target potential could be controlled to below 1 mV if K_P is set to an appropriate value.

Key words: potentiostat; PC; phase analysis of galvano-chemistry

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Potentiostats have many uses in the phase analysis of galvano-chemistry, such as electrolysis under constant potential, electrolysis using galvanostatic method, electrolysis in constant quantity of electricity, determination of polarization curves and the etching quantitative phase analysis (scanning potential method, step current and step potential method) *etc.*[1–3]. Different kinds of apparatus are needed for different experimental purposes, thus polarization conditions are different which are required in the galvano-chemistry experiments and the relative methods of measuring and analyzing the experimental results. However, rapid development and wide application of the microcomputer technology have made it possible to do those different experiments only by using a usual constant-potential power supply together with a PC. A device designed in this paper comprises of a PC, one A/D & D/A converter and relative program running in the PC. It could be used not only for electrolysis in constant potential and determination of the *E–I* polarization curve, but for electrolysis in constant quantity of electricity, measurement of the etching quantity of electricity under constant or dynamic potential, determination of the *E–lg I* curve, control of the complex polarization behavior, thus development and application of the phase analysis of galnovachemistry.

1 Components of the device

The measurement and control of the device is shown in the **figure 1**. An A/D & D/A converter used in the device is called 818HD (12 bits) which is manufactured by Evoc Company. A digital amplifier, Evoc 789D, is used to amplify the fore-end signal. Its internal resistance value to 200 MΩ and its enlargement factor is 3, which can satisfy the requirement of measuring accuracy of electrode potential. A remote regulated direct-current power supply DH1719 is used to compensate for the low output power of the D/A conversion. The maximum output voltage of DH1719 is 20 V and its power is 5 W.

The operating principle of the device is described as following. (1) The sample acts as the anode in an electrolysis system. (2) The potential E_I between the sample and the reference electrode is imported into a PC, being converted to digital signal by the A/D converter first. (3) The program running in the PC calculates the con-

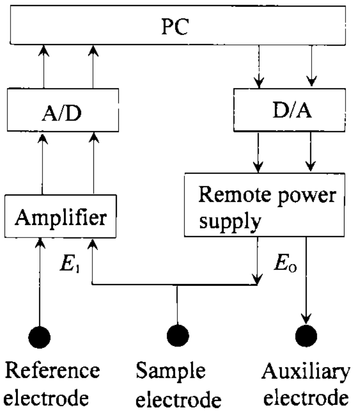

Figure 1 Schematic diagram of measurement and control.

trol variation e according to the target control potential E_D, then exports the voltage calculated according to a proportion factor K_P to control the potential between the sample and the auxiliary electrode *via* a remote power supply. The process above is repeated until the actual anode potential equals or nearly equals to the target control potential, *i.e.*, the control accuracy is reached.

In order to determine the current density, a resistance could be concatenated in the device whose voltage could be used to calculate the current. Then the current density could be calculated according to the surface area of the anode.

2 Algorithm of controlling the electrode potential

The traditional potentiostat is a kind of differential amplifier with negative feedback [1,2]. Its working principal is as follows: Its output voltage is sent back to its input port. Since the feedback signal has opposite phase to the original one, the two signals are superposed and then amplified by the amplifier. Finally, a stabilized output voltage could be obtained.

While the traditional potentiostat realizes measurement and control through certain electric circuit, the device designed learns itself and implements the control through software.

The controlling algorithm of output voltage is shown as

$$\Delta E_O = K_P\, e,$$

where ΔE_O is the variation of output voltage, K_P the proportion factor, and e the difference between the measured anode electrode potential E_I and the target control potential E_D.

The control in the device equals to a kind of proportional controller. A suitable K_P leads to a satisfying result of control.

The software is programmed in Turbo C 2.0 in order to run in DOS environment and thus decrease the hardware cost of the device. The diagram of the program is shown in **figure 2**. Polling routine is used in the program through which the potential difference between the sample electrode and the reference one could be input through A/D conversion. Then the program calculates the output voltage based on certain parameters and transfers the signal to the remote power supply, DH1719. Finally, the amplified output voltage is output to the sample electrode and the auxiliary one. In the program, the modules of function generation and the logarithmic transformation of current are designed to replace the function generator and the logarithmic transformer in the traditional potentiostat.

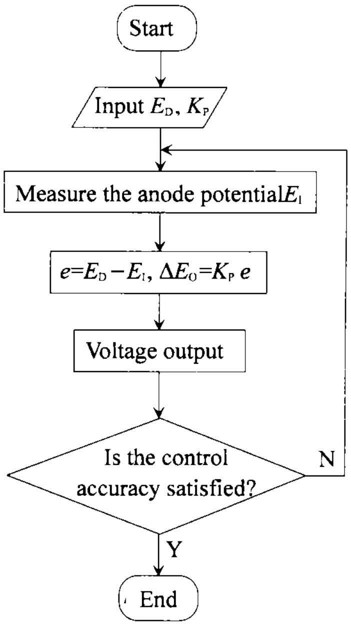

Figure 2 Flow chart of the program.

3 Experimental results and evaluation of the device

The measured electrode potential and current can be stored as ASCII in the PC, which can also be used for further analysis by some application software when needed, like Origin, Excel and so on. Two experiments using the device, the electrolysis in constant potential and the determination of the polarization curve, are described in details as follows.

3.1 Control of electrolysis in constant potential

The electrolyte in the experiment is 10% AA solution, the reference electrode is calomel electrode, and the anode is X52 steel (micro-alloyed steel, fractured at 900℃). The purpose of the experiment is to electrolyze the precipitate of vanadium in the X52 steel. According to the reference [1], the anode potential is -100—-200 mV vs. SCE. In the experiment, the anode potential is controlled at -150 mV vs. SCE.

The electrolyzing cell is a quite complex reaction system where the coulometric transfer, galvano-chemistry variation, and the change of compositional density all take place. This made it rather difficult to analyze the electrolyzing cell. Moreover, its equivalent circuit is usually used for qualitative analysis and semiquantitative assessment. If we treat it as a black box, however, its internal mechanism will be ignored and its basic parameter, the anode potential, will just be the object of consideration and control.

In fact, the essence of the device is to take advantage of PC as a proportional controller to control the electrode potential. In the system, K_P is the setting parameter. The smaller K_P is, the more easily the system becomes stable while the larger the steady-state error is. The effect of K_P on the variation e in the process of control of the potential is shown in **figure 3**, from which it can be seen that there exists an extreme value for e and e arrives at its minimum when K_P equals to 0.01. As is known, when K_P is smaller, the system is more stable and the variation is chiefly caused by the steady-state error; and when K_P is larger, the variation is mostly caused by the instability of the system. In the system, it is suitable to set K_P to 0.01 and then the variation e is 1.66 mV that could satisfy control requirement of the constant potential. It indicates that the device designed can replace the traditional potentiostat and be applied in the measurement and control under constant potential.

3.2 Determination of the polarization curve

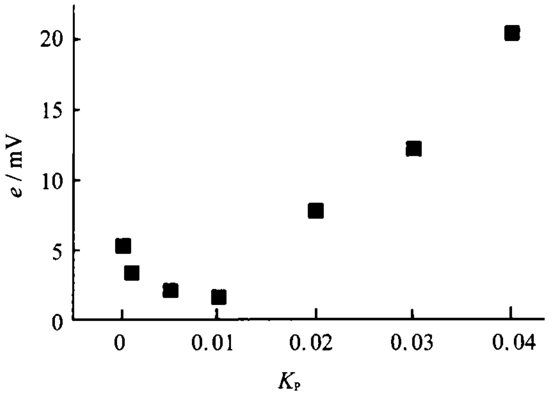

Figure 3 Effect of the enlargement factor on the average variation.

The electrolyte, the reference electrode and the anode in this experiment are the same as the ones in the experiment above-mentioned.

The variation of the target potential and the actual potential with the time are shown in **figure 4**. Because the average variation between the controlled potential and the target one is about 1 mV, the system can be used for determination of the polarization curve.

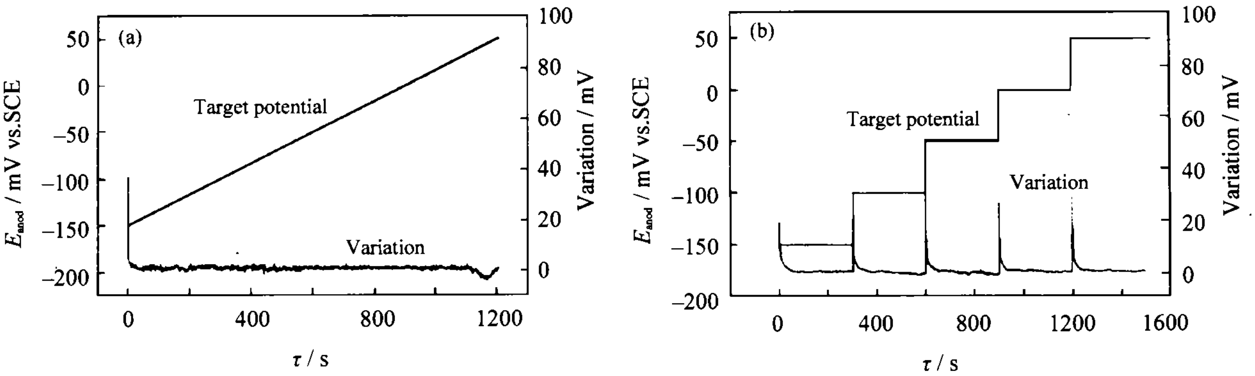

Figure 4 Changes of the target potential and the average variation with time in the way of scanning where the rate is 10 mV/min (a) and in the way of step poteutial where the time interval is 50 mV / 5 min (b).

4 Conclusions

(1) The variation between the actual anode potential and the target potential could be controlled below 1 mV if K_P is set to a suitable value.

(2) The device designed can substitute for the traditional potentiostat in the control of constant potential and the determination of polarization curves. Accordingly, function generators, X-Y recorders and traditional potentiostats are not needed for these experiments.

(3) Compared with traditional potentiostats, the device designed has several advantages, such as easily operated, easy to process data, and high accuracy of control and measurement, *etc.*. Further more, many kinds of control in the phase analysis of galvano-chemistry could be realized only through modifying the PC program in the system.

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