Platinization and Microelectrode Impedance Monitoring by Internet

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Abstract

The use of the Internet is applied in this work in order to remotely monitor and control an experiment. The electroplatinization of microelectrodes and subsequent impedance monitoring is performed.
Important variables such as response time and package lost are analyzed here and characterized for performing this kind of experiment.
By means of TCP/IP protocol evaluation it is shown that the Internet is able to transfer 1kb packages within less than a second to places as far as Germany, and so by combining the aspects of a "de facto" experimental setup (server) and remote control and monitoring (client) this work proposes a new implementation of the Virtual Laboratory.

1. Introduction

For quite a long time metal microelectrodes have been used to measure action potentials, extracellular potentials and nervous cells potentials, however there are still open questions on how their shape might be, which material would be more suitable for their development, how to combine them in arrays and how to interpret signals obtained from them [1], [2].
This work presents the characterization of some electrical properties, especially the impedance measured in a metal microelectrode immersed in saline solution. According to Warburg [3], [4], this impedance is formed by the series association of the capacitance found in the interface between metal and electrolyte and by the microelectrode resistance itself.
According to Geddes [5], this impedance can be estimated by the following equation (1):

$$Z = \frac{0{,}225 \cdot 10^{14}}{K \cdot a \cdot f^{(1-a)}}, \quad (1)$$

where Z is the impedance in ohms, K is the capacitance/cm^2 coefficient of the interface metal-electrolyte, a stands for the electrode area in μm^2, α is the capacitance coefficient which varies according to the frequency, and f the frequency in Hertz.

Using a physiological saline solution (0,9% NaCl) as electrolyte and platinum black as electrode, the result will be K=4950 and α=0,366 [6]. In other words, the impedance tends to fall when the frequency and the electrode area increase. Impedance must be low enough in order to be measured by the provided instruments. Therefore, the process of platinum black deposition electrodes will be used in order to increase their area. In order to obtain an impedance of about 50kΩ with a 1kHz frequency, the electrode area should be larger than 1100μm^2.
It is desired that both the platinization process and the microelectrodes impedance measurement are performed using Internet. Thus, the concept of Virtual Laboratory [8], [9] is used to propose a method of receiving and sending any kind of experimental data through computer networks.
Due to the applications for Virtual Laboratory, this concept is often associated to Virtual Instrumentation and Internet. By using Virtual Laboratory, it becomes possible to setup and remotely control instruments which have a GPIB interface. Figure 1 shows the basic scheme of a Virtual Laboratory where data of an experiment are sent by a computer located in the laboratory, where the experiment occurs, to a remote computer, by Internet.

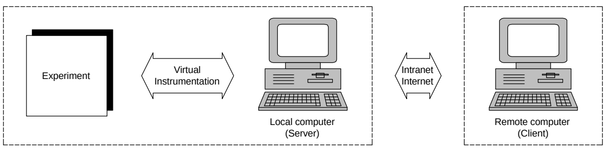

Fig. 1: Basic scheme of a Virtual Laboratory

2. Materials and Methods

A microstructure containing 100 square electrodes metallized in gold was used for the experiments. Electrodes had superficial area of 400μm^2 and enclosed a total area of 1mm^2. The manufacturing process of this microelectrode array and its application are presented in [6] and [7]. Figure 2 shows an example of the used array.

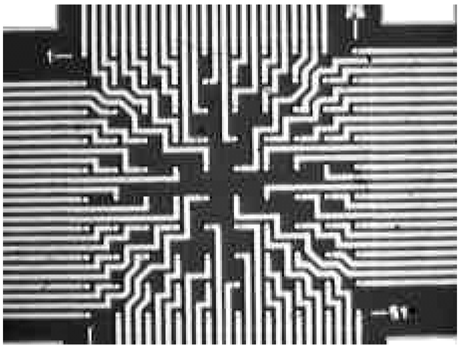

Fig. 2: Display of the manufactured microelectrodes array central area (1mm^2 in the picture), with 120µm space between adjacent electrodes.

As previously seen, the area of microelectrodes must be increased by means of an electroplatinization process in such a way that impedance decreases to values which may be safely measured by the available equipment. The platinum black electrodeposition is performed using a H_2PtCl_6 solution and a current density which may vary between 100 and 500mA/cm^2. This current is limited by a 1,6MΩ resistance in series with the electrode.

To accomplish both the platinization process and the measurement of the electrodes impedance simultaneously, an experimental apparatus was setup as shown in figure 3.

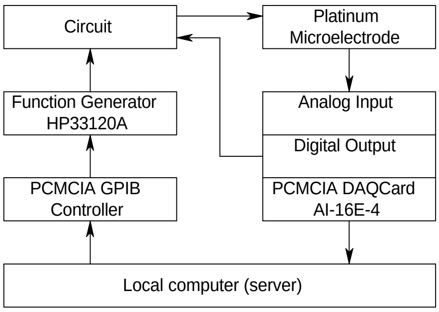

Fig. 3: Setup block diagram.

DC voltage (5V) was supplied by a Hewlett Packard function generator HP33120A [10], which provided also a 100mVpp (1kHz) sine signal, which was used for the microelectrode impedance characterization. The DAQCard AI-16E-4 (data acquisition card) from National Instruments [11] measured voltage on a 100kΩ reference resistance connected in series with the microelectrode, in order to evaluate voltage, current and microelectrode impedance. A notebook was used to control both the function generator through the GPIB interface, and the DAC.

During platinization, current has to be limited to 3µA so that no damage is done on electrode metallization. The circuit shown in figure 4 controls maximum current. It contains one switch, controlled by one of the DAC digital outputs, which forces current through the 1,6MΩ resistance connected in series to the microelectrode (besides the 100kΩ reference resistance itself).

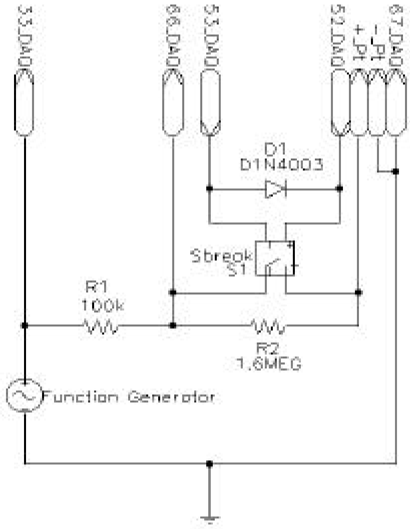

Fig. 4: Circuit diagram for current limiting during platinization.

All the process is controlled by means of a VI (Virtual Instrument) created in LabVIEW 5.1 - National Instruments [13]. This VI, called *Platinização e Medida de Impedância - Servidor.vi,* is also responsible for data transmission by Internet. Another VI, named *Platinização e Medida de Impedância - Cliente.v*i, is executed in a remote computer in order to start platinization process and display the microelectrode impedance values as a function of time.

The VI *Platinização e Medida de Impedância - Servidor.vi* has its flowchart shown in figure 5 and can be described by the following steps:

i) Wait for a TCP/IP connection.
ii) Read through the established connection the parameters to be used, defined in the remote computer as *Platinização* (it selects if platinization will occur before measuring the impedance), *Tensão Pt.* (voltage to be used during the platinization), *Dt Pt.* (duration of the voltage pulse that platinize the microelectrode), *Amplitude Sn* (sinusoidal signal amplitude), *Dt Sn.* (sinusoidal signal duration), *Freqüência Sn.* (sinusoidal signal frequency), *Rref* (reference resistance real value of the that is being) and *Salvar Dados* (it selects if measured data will be saved or not).
iii) Open switch S1, limiting platinization current.
iv) Execute platinization process, controlling voltage on function generator output.
v) Close the switch S1.
vi) Calculate the microelectrode impedance value based on the DAC measurement voltage of the reference resistance and transmit it to the remote computer.
vii) Microelectrode platinization process continues until impedance value becomes smaller than *Zmáx* defined by user and halts if the impedance value is smaller than *Zmín*, all the process will be interrupted in order to prevent microelectrode damage.

viii) Close TCP/IP connection.

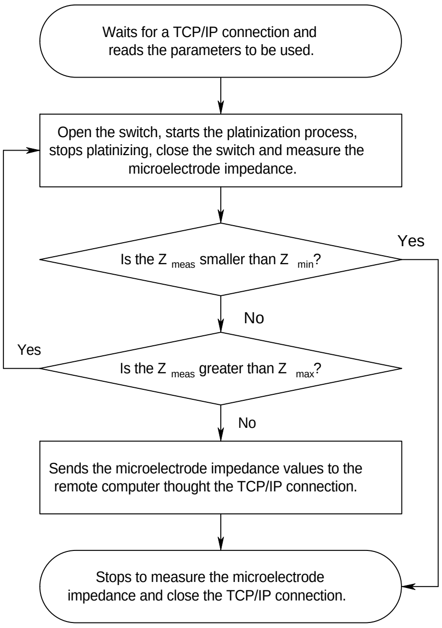

Fig. 5: Flowchart of the VI "Platinização e Medida de Impedância - Servidor.vi".

The flowchart of *Platinização e Medida de Impedância - Cliente.v*i is shown in Fig. 6 and can be described as it follows:

i) Open TCP/IP connection with server.
ii) Send parameters to be monitored and controlled.
iii) Show impedance values of microelectrode measured.
iv) Close TCP/IP connection.

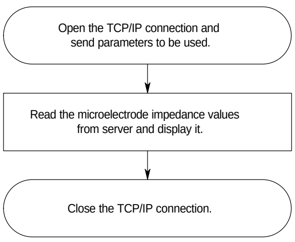

Fig. 6: Flowchart of the VI "Platinização e Medida de Impedância - Cliente.vi".

In order to characterize medium response time through the network, measurements were performed and the traffic level in Internet was monitored during a working day with three paths covering different distances. Location of each computer pair was chosen as follows:

1. Two computers at the Microelectronics Laboratory from the State University of São Paulo (USP);
2. A computer at the State University of Campinas (Unicamp) and another at USP;
3. A computer in the University of Bonn, Germany and one at USP.

Ping Plotter 2.03 was setup for TCP/IP protocol sending 1024 bytes packages, waiting for 10s (maximum timeout), and 60s pause between two packages.

3. Results and Discussion

Analyzing microelectrode array after the platinization process (Fig. 7), one can verify that it was not possible to do the platinization in every microelectrode because of the interrupted tracks. It was also noted that some microelectrodes were damaged due to excess platinizing current which passed through them, this probably happened as a consequence of response time differences found in output signal variation of the function generator and in the switch activation.

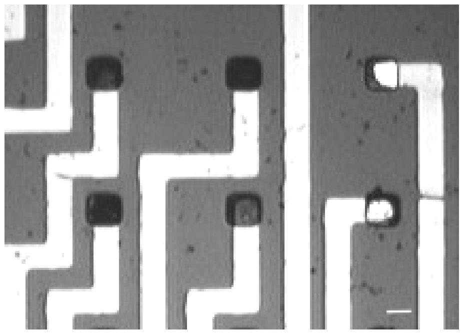

Fig. 7: Visual evaluation of track and electrode state, passivation and platinization (image from the optical microscope).

Furthermore, one can analyze platinum deposition by observing the derivative of impedance value function, as it can be seen in Fig. 8. When the process comes to the end, there is no sudden variation anymore.

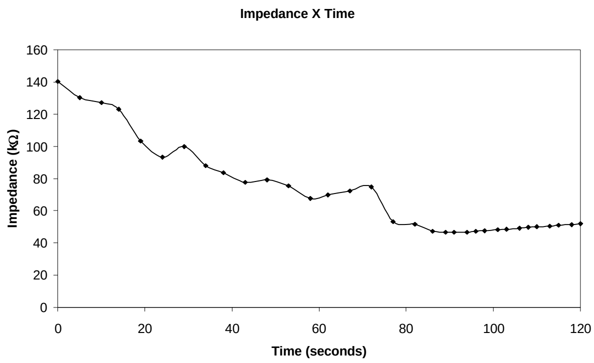

Fig. 8: Microelectrode impedance variation as a function of time.

As far as the data transmission by Internet is concerned, the following results were obtained:

Path		Average ping response time (ms)	Lost packages
USP	USP	4,2±2,5	0,09%
	Unicamp	221±226	22,1%
	Univ. of Bonn	727±495	29,5%

Table 1: Ping response times and lost packages percentage calculated.

In Fig. 9, 10 and 11 traffic evolution in Internet during a period of 24 hours may be observed and compared among the three computer pair instances used.

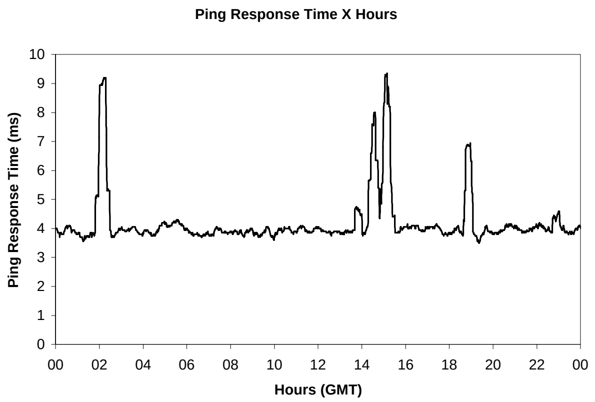

Fig. 9: Graphic showing ping response time of 1024 bytes-packages between two computers placed at the Microelectronics Laboratory, at USP.

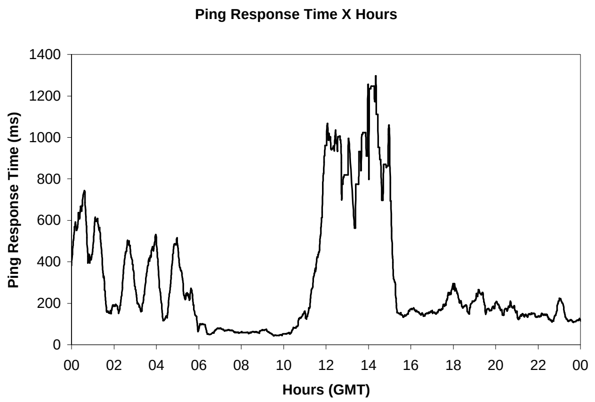

Fig. 10: Graphic showing ping response time of 1024 bytes-packages between a computer located at USP and another at Unicamp.

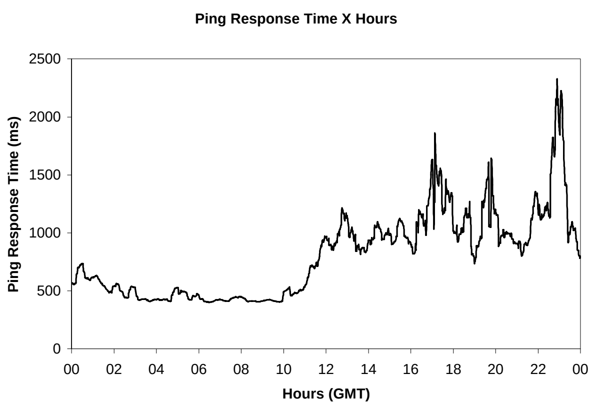

Fig. 11: Graphic showing ping response time of 1024 bytes-packages between a computer located at USP and another at the University of Bonn, Germany.

4. Conclusions

Through the obtained results, it is possible to conclude that the current infrastructure of Internet allows one to remotely perform experiments if response time is not a critical variable. The reply time and amount of lost packages between two distant computers will be smaller if there are less routers between them.
Depending on the implemented application, the use of some feature that guarantees the security of the process may be necessary. In this experiment, the program stops the platinization process automatically, in the case that the possibility of damage to the microelectrode is detected.

5. Acknowledgments

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