

Realization of the Kelvin Probe System for the Surface Treatment of a Semiconductor

C. Zegadi^{1,*}, Z. Lounis², A. Haichour^{1,†}, A. Hadj Kaddour², D. Ghaffor²

¹ *Micro and Nanophysics Laboratory (LaMiN), National Polytechnic School of Oran, ENP Oran-Maurice AUDIN, BP1523 El-Mnaouer, 31000 Oran, Algeria*

² *LABMAT, National Polytechnic School of Oran, ENP Oran Maurice AUDIN, Oran, Algeria*

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The knowledge of the electrical properties of materials is inevitable in surface technologies, such as microtechnology, corrosion, etc. Concerning the surface phenomena, the work function represents the main property. It was developed by Lord Kelvin and it corresponds to the contact potential difference between two surfaces of materials. In this project, the data acquisition of Kelvin Probe System (KPS) was performed after sequential tests in electronic computing and physical fields in order to acquire the work function of conductor and semiconductor materials. This system has revealed the great importance of controlling the support voltage V_b calculating the capacitor applied to the Metal-Insulator-Semiconductor (MIS) structure in order to measure the surface potential of the semiconductors. Some problems were solved during the assembly of the system and the pertinent frequency of 50 Hz was suitably adjusted. However, the conversion of current-voltage was not carried out in KPS due to the insensitivity of the amplifiers on hand. To understand this difficulty in signal experimental study, we have used a calculation by a Fortran code. The latter has confirmed that the signal of Kelvin probe is a very weak amplitude of the order of pico-volts. Because of the available measuring devices whose sensitivity is much lower than the signal itself, on the other hand, these results justify the experimental steps.

Keywords: Contact potential difference, Kelvin probe, MIS structure, Surface phenomena.

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1. INTRODUCTION

Several surface proprieties are deduced by the work function, namely the reconstruction of surfaces, doping and band bending of semiconductors, trapping of charges in dielectrics, catalytic activity, and corrosion. Recently, the work function map produced by Kelvin probe force microscopy (KPFM) has given useful information on the surface of a solid [1], on its chemical composition and their electronic state of the local structures. So, it has become an alternative to non-contact scanning probe microscopy (SPM) which avoided several difficulties for the latter like the nature and the fragility of the cantilever and the positioning of the laser on the cantilever [2]. However, the Kelvin probe method consists in creating a simple capacitor between a circular plane electrode and the specimen [3, 4]. To prevent a single occurrence, a circulation of the charge, a bit of vibration to change of the ability was used that thereby allowed the circulation of the charge to be renewed. In this project, we report the most important steps for the Kelvin probe assembly, the tests which were carried out to check its effectiveness, with the citing the difficulties which hindered the reading of a very low signal. The difficulties were overcome by explanation using a simulation with a Fortran code.

2. RECALL OF PRINCIPLE WORK IN KELVIN PROBE

Pursuant to physical principles of a Kelvin probe, we refer to the three methods (a, b, and c) shown in Fig. 1. Here Φ_1 , Φ_2 are the different energy diagrams of electrons of the two specimens and are the work functions of

materials, E_1 and E_2 represent their Fermi levels. The third method gives us the difference work function W_f between the two surfaces, the only point where the electric field between the plates disappears, W_f is the same as a "support potential" V_b . Based on the latter, Fig. 2 shows an instrument of "Scanning Kelvin Probe Arrangement" [3]. A synthetic digital sine wave form applied to the coil allows the computer to control oscillation frequency, amplitude, and pitch. The probe is directly mounted on an I/V converter. Both the sample and the probe are connected via a voltage source of support potential V_b , which is controlled by a 16-bit digital to analog converter (DAC). A theoretical calculation of the signal will be carried out according to the equivalent scheme of the Kelvin probe in Fig. 2 [3, 4].

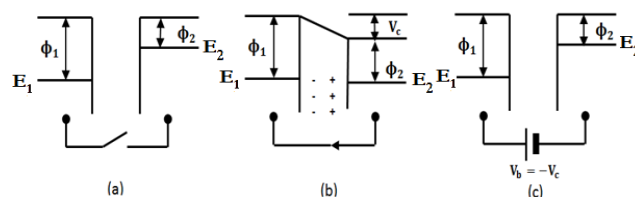


Fig. 1 – Electron energy level diagrams of two different metals: (a) non-contact, (b) with electrical contacts, (c) support potential

2.1 Signal Calculation

Using a vibration probe, the production of capacity is given by:

$$C_X = Q/V = \epsilon_r \cdot \epsilon_0 \cdot A / d(t), \quad (2.1)$$

*chawki.zegadi@enp-oran.dz
†haichour_amel@hotmail.fr

where Q is the charge, C_x is the capacitance, A is the surface of the capacitor, d is the distance between the plates, and ϵ_0 is the dielectric permittivity. The intensity (i) of the circuit is written as:

$$i = dQ / dt = (V_c + V_b) \cdot dC_x / dt, \quad (2.2)$$

where V_c is the contact potential (the capacitor), V_b is the support potential. The relation between V_{in} and V_{out} is as follows (see Fig. 2 and Fig. 3a):

$$V_{out} = iR + V_{in}. \quad (2.3)$$

The movement $d(t)$ of the tip is given by:

$$d(t) = d_0 + d_1 \cos(\omega t), \quad (2.4)$$

where d_0 is the average distance between the sample and the end of the probe, d_1 is the oscillation amplitude of the probe; t is the time and ω is the angular pulse [6, 7]. So, we obtain V_{out} as a function of V_b , V_c , A , ϵ_0 , d_0 , d_1 and t (Eqs. (2.1)-(2.4)):

$$V_{out} = -(V_c + V_b) \cdot R \cdot A \cdot \epsilon_r \cdot \epsilon_0 \times d(1/d_0 + d_1 \cos(\omega t)) / dt \quad (2.5)$$

3. IMPORTANT COMPONENTS OF THE KELVIN PROBE EXPERIMENT

In order to facilitate access to the external environment, it was interesting to use a parallel port of the computer. The system for producing electronic cards often requires the addition of software via an input/output port of the computer. The different addresses have been programmed using the Delphi language, which allows creating graphical interfaces. We tried to build the proper system to the Kelvin probe experiment, where the work was shared in two parts: 1 – the electronics and information technology (IT) part; 2 – the second part will be devoted to the physical phenomena.

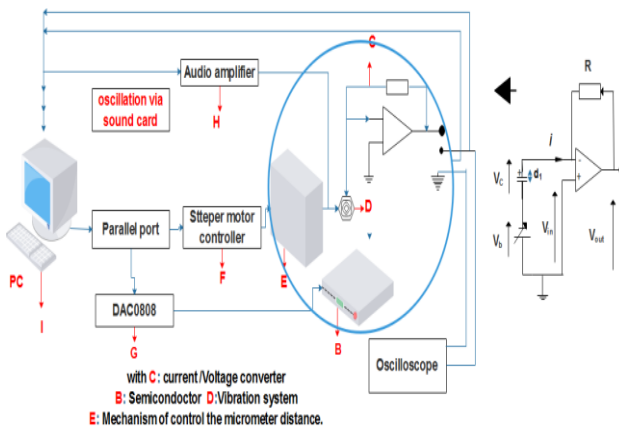


Fig. 2 – System of the Kelvin probe experiment realization

3.1 Electronics-information Technology (IT) Instruments in Kelvin probe

3.1.1 Parallel Port

The parallel port is associated with Centronics Parallel Interface. It was designed for a text printer that uses an 8-bit character set and reads and writes data

via the parallel port of the status port in "Delphi" under WinXp [8]. In this work, the unipolar stepper motor is chosen [9]. It has six queues in two triples and each triplet feeds a coil whose center is common (two separate spools). To turn a stepper motor, the coils were fed, two by two each time. There are always four steps. The first step is to connect the two common terminals to the supply voltage. Then the four coils were fed in sequence.

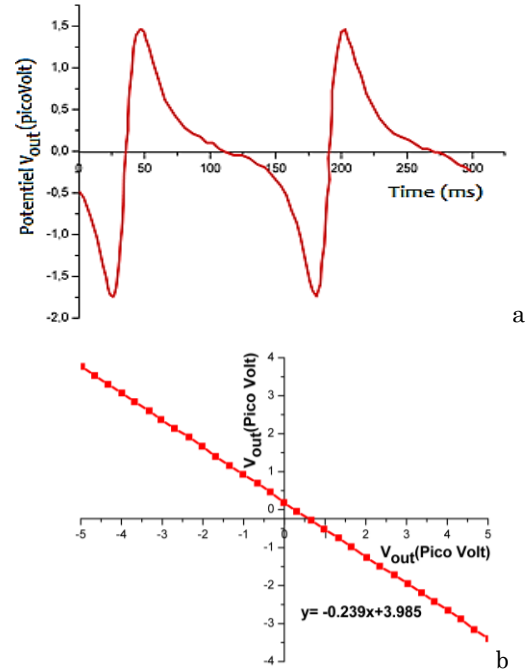


Fig. 3 – (a) Kelvin probe signal over time; (b) V_{pp} -peak to peak voltage

3.1.2 Stepper Motor Displacement

The motor control allows the carriage movement. This mechanism governs small distances of the order of a micrometer for this. Simple means have been investigated having the following characteristics:

- Stepper motor: unipolar type of 48 steps/tour.
- Rotation axis: small thread of 0.5 mm and 6 mm diameter.
- Equilibrium system: four diagonal rods to the rotation axis.
- Pulley: 384 steps/tour and for short distances we used the half-step mode, so $384 \times 2 = 788$ steps/tour.

3.1.3 Control Interface for Two Parallel Motors

The model was used to operate two unipolar stepper motors from the parallel port of the PC. The signals that drive each rotating motor are delivered directly by the eight data lines of the output port which is a hardware configuration convenient enough for most of the work to be carried over to programming.

3.1.4 The Digital/Analog Converter (DAC0808)

The digital-analog converter (DAC) is an electronic component [10]. It allows the transformation of a physical quantity (voltage, current) into a numerical value. The control of the DAC by PC is given by:

$$V_{out} = V_c \times (a_1 / 2 + a_2 / 4 + a_3 / 8 + a_4 / 16), \quad (3.1)$$

where a_1, a_2, a_3, a_4 are the binary values equal to 0 or 1. The results for the support voltage ($V_{out} = V_b$) in binary system are shown in Table 1. So, we get 15 values of the output voltage V_{out} between 0-4.6875 with a step of 0.3125 V.

Table 1 – Description of the special paragraph styles

a_4	0	0	0	0	0	0	0	0	1	1	1	1	1	1	1	1
a_3	0	0	0	0	1	1	1	1	0	0	0	0	1	1	1	1
a_2	0	0	1	1	0	0	1	1	0	0	1	1	0	0	1	1
a_1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1
Tension (mV)	0	0.3125	0.6250	0.9375	1.2500	1.5625	1.8750	2.1875	2.5000	2.8125	3.1250	3.4375	3.7500	4.0625	4.3750	4.6875

3.1.6 Current/Voltage Converter

We made use of our circuit of a digital system operational amplifier with accuracy voltage current converter. It is sufficient that the parallel impedance of the power source is large compared to R . The voltage is then:

$$V_s = -R \times I_m. \quad (3.2)$$

The amplifier is used to increase the low-amplitude signals and to improve the signal/noise ratio and there-

3.1.5 Diagram of the Electronic Interface that Controls the Two Stepper Motors

Among the existing solutions to generate an analog signal from a digital system, the DAC0808 was chosen to work with. For the control of the DAC, we associated the 4 pins of motor 2 of the parallel port with the 4 pins of the DAC0808. Fig. 4 represents the interface of the electronic circuit.

fore to render exploitable information.

The gain value is calculated in such a way that the current will be of the order of pico-amps to improve the accuracy of the measurement. The LM358 Ampli-Op is used to convert the current supplied into voltage when it is connected to high resistance. Then, it is interesting to use the latter to obtain a 50 Hz signal. The achievement of this result prompted us to reduce the noise and the electric field by three techniques in order to eliminate the parasitic signal of 50 Hz [11].

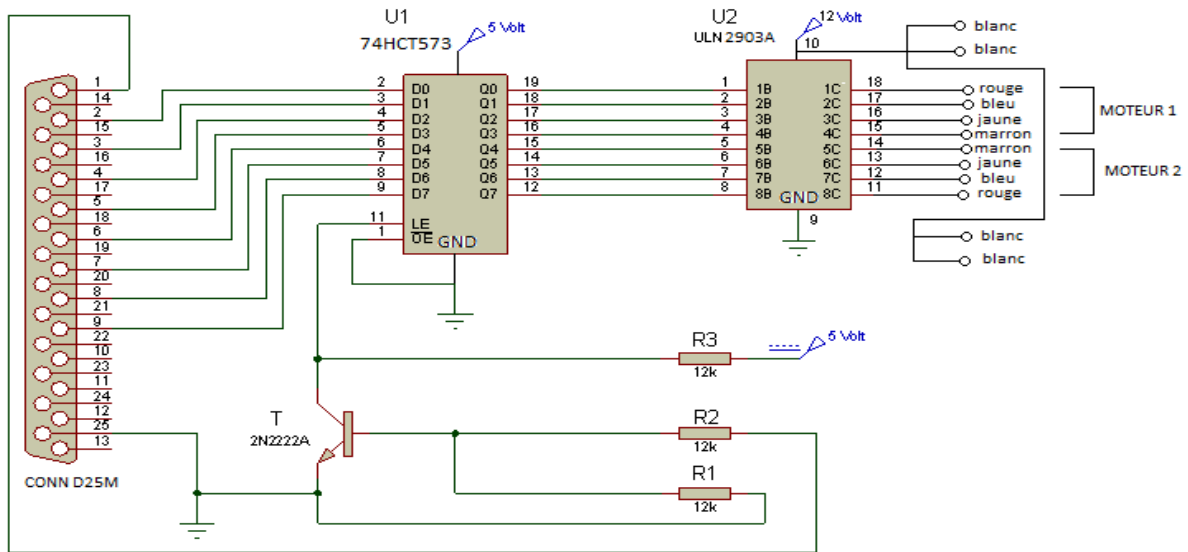


Fig. 4 – The interface of the electronic circuit obtained

3.1.7 The Anti-aliasing Filter and the Faraday Cage

Filters are electrical circuits whose main property is to allow certain useful frequencies to pass through and to block others, such as coaxial cable. We have used a Faraday cage of dimensions: 50 cm long, 30 cm wide, with internal and external coating, respectively, by aluminum and an insulator.

Electromagnetic shielding is a protection against parasitic signals. This protection is generally inspired by the Faraday cage; it forms a conductive envelope where the electrical and electromagnetic parasitic signals are

routed to the ground. The thickness of the shield has only a mechanical resistance effect, and it does not affect the quality of the protection.

4. PHYSICAL TEST

4.1 Realization of the Probe

The probe preparation method has been tried to be exposed in our physical tests, that is why two probes of different forms have been prepared:

- The first test: an indium metal ball is taken as a probe (diameter 1 mm and work function 5 eV).

– The second test: a plane copper plate (surface 26 mm² and work function 4.71 eV).

In order to paste them on a conductive surface, the practical steps of the "lithography and sputtering techniques" performed both in the two-probe experiment will be shown and explained.

4.1.1 Photolithography and Metallization

The step of the photolithography consumed in this work is exposed, which consists in the application of a photo-resin in the form of a thin film on the surface of a substrate. It is then exposed to light radiation during this step. The use of a mask, formed of opaque and transparent zones, makes it possible to define the pattern which is desired to be reproduced on the substrates [12].

After the development of the resin, it is necessary to metalize certain areas of the substrate. To this end, we have deposited the nickel on our areas by RF sputtering. After deposition of nickel, we get rid of the insolated resin to have only the negative part of the mask as a thin layer pattern on the substrate. In our case, we used acetone as a developer.

4.2 Study of the Capacity

4.2.1 Study of the Capacity: Metal Ball-Insulator-Semiconductor

For the measurement of the capacity of the MIS structure (metal-air-semiconductor), two experiments were realized with two different types of metals (an indium ball and a flat copper surface) and with the same type of semiconductor Cu₂S (*p*) [4].

Note that *C* is the capacity measured between a metal and a semiconductor; it is of the order of picofarad. The distance *d* is given by:

$$d = N \times 1.3 \tag{4.1}$$

with *N* = number of steps and 1.3 μm displacement of the metal surface equivalent to one step. These results are represented in the graph of Fig. 5.

4.2.2 Study of the Capacity: Flat Surface Insulator-Semiconductor

For the second experiment, a flat copper surface was used; the results are shown in Fig. 6. The red curve represents the fitting of the results obtained and also shows the variation of the capacitance *C* in the form of 1/*d* of the Eq. (2.1). We note that the two curves in Fig. 5 and Fig. 6 reflect the same shape of the theoretical expression of the capacitance of a planar capacitor but with deviations from the adjustment which can be explained perhaps by several influencing factors, namely measurement errors (displacement uncertainty, capacitance meter), the nature of the materials, ex situ of the experiment or the dielectric permittivity of the medium $\epsilon_r = 1$ in dry air, which varies with the ambient conditions (humidity, pressure, temperature, etc).

However, in this study, the capacitor in both experiments varies inversely with distance *d*. This leads us to conclude that the dimension of a ball is mostly large

compared to the distance between the ball and the substrate and the formula $C = \epsilon_0 \times A/d$ of the plane capacitor remains valid in the ball case.

Conversely, for a small dimension of a ball, the equation of the capacity will be given by the formula $C = 4 \times \epsilon_0 \times R$ with the radius of the sphere *R*.

Table 2 – Capacity measurement results for the first experiment

<i>C</i> (pF)	Number of steps	<i>d</i> (μm)	1/ <i>d</i>
10.9	6	7.8	0.12800
10.7	12	15.6	0.06400
10.6	18	23.4	0.04270
10.5	24	31.2	0.03205
10.4	30	39	0.02564

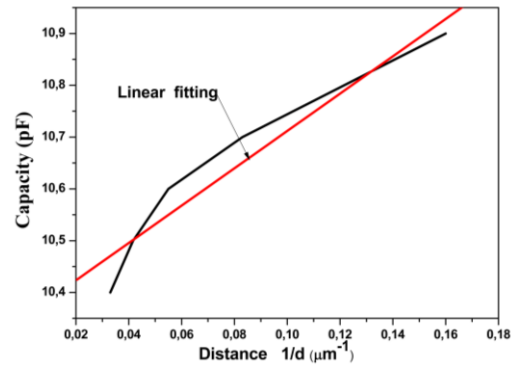


Fig. 5 – Capacity variation as a function of 1/*d*

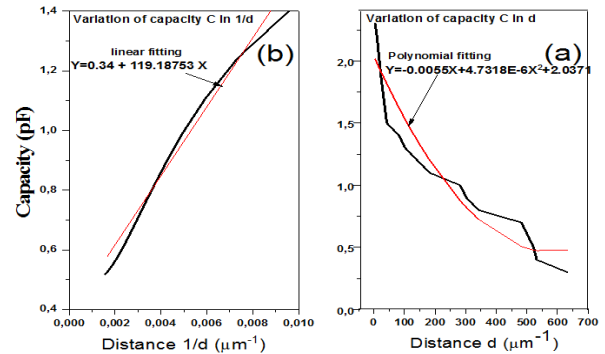


Fig. 6 – (a) Variation of the capacity as a function of *d* (the distance between the copper surface and the substrate); (b) Variation of the capacity according to 1/*d* and results of the fitting (red color) for the plane copper surface

5. SIMULATION TEST

This part is devoted to the study of different physical phenomena for the MIS structure such as the capacitance and the difference in contact potential. We carried out this simulation in order to check the agreement between the results obtained by our realized system (the Kelvin probe) and the model programmed on the plane surfaces. In this test, we determine the signal *V_{PP}* given by the following equation (see Fig. 3b), using as input values the capacitance *C* measured experimentally and determined previously with *d* – the distance separating the ball from the sample [13]:

$$V_{out} = \frac{RC\omega A \varepsilon_0 \varepsilon_r (V_b + V_c)}{d_A} \cdot \frac{\sin(\omega t)}{\left[1 + \frac{d_1}{d_0} \cos(\omega t)\right]^2}. \quad (5.1)$$

To understand the problems encountered in the experimental study, a small simple program written in a Fortran language that simulates the signal was used. In this simulation, the parameters of the vibration system (the lens of the CD player) were used which allowed to see an electrical signal that varies according to omega. These parameters are: the flat copper surface $A = 26 \text{ mm}^2$, the distance d_0 equal to $135.4 \text{ }\mu\text{m}$ and the value of the capacitance equal to $1.25 \times 10^{-12} \text{ F}$. From the graph in Fig. 6 and by replacing these values in Eq. (5.1), the product of the dielectric constants is equal to $6.5 \times 10^{-12} \text{ F}$, so it can be said that the work was done in the center close to the blank = $8.85 \times 10^{-12} \text{ F}$.

In this work, the variation of the support potential V_b between 0 and 15 V and the vibration frequency of the copper surface between 50 and 800 Hz using Win-Oscillo were taken. With these parameters [13], the result of the V_{out} voltage measurement is not obtained. So, V_c voltage equal to 0.33 V was taken. According to the simulation data, the vibration amplitude is equal to $3.5372144 \times 10^{-12} \text{ V}$ and the peak crest voltage is about $7.074 \times 10^{-12} \text{ V}$. The graph obtained is shown in Fig. 7.

We have to note that first, resistance $R = 10 \text{ M}\Omega$ was chosen to work with. This value did not allow to display the signal obtained in the simulation. So, resistance of a very high value ($10 \text{ G}\Omega$) was more convenient, and a signal was obtained that was visualized on the oscilloscope but whose value is very low [5].

6. CONCLUSIONS

The technique of Kelvin probe is non-destructive, minimally invasive and offers an attractive method to obtain high-resolution maps of the surface potential distribution on conductive and non-conductive samples. For that reason, we have tried to build the appropriate system for the Kelvin probe experiment and we made it based on two parts: electronics-information technology (IT) and physics. In this context, we have devoted to study the different physical phenomena for the MIS (metal-insulator-semiconductor) structure such as the

capacitance and the contact potential difference. In order to measure the capacity of the MIS structure, two experiments have been performed through an indium ball and a copper surface chosen as metals whose exit energies are known. These metallic elements have been incorporated into Kelvin probe via both physical ways used for the development of thin layers: lithography and sputtering techniques. The physical results demonstrated that the capacity of these two probes is similar to the planar capacity. Furthermore, we have carried out a simulation in order to verify the agreement between the results obtained by our realized system (Kelvin probe) and the model programmed on the plane surfaces. In this test, we have checked the signal V_{out} given by the theoretical equation. The result of the Fortran simulation confirmed and revealed that the signal got from Kelvin probe is very weak, the amplitude of which is around pico volts. For this reason, our measuring devices used could not detect it, where their sensitivity is much lower than the signal itself (sensitivity of 0.1 mV). On the other hand, the results obtained validate the experimental stages of a successful assembly of Kelvin probe.

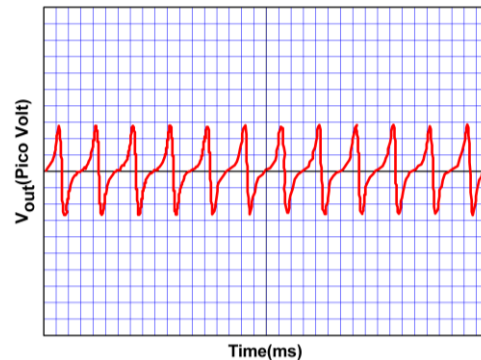


Fig. 7 – The graph of the voltage V_{out} using a simulation

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REFERENCES

1. P. Eaton., P. West, *Atomic Force Microscopy* (Oxford University Press: Oxford, U.K.: 2010).
2. S. Sadewasser, Th. Glatzel, *Kelvin Probe Force Microscopy, Measuring and Compensating Electrostatic Forces* (Verlag Berlin Heidelberg, Springer: 2012).
3. I.D. Baikie, P.J. Estrup, *Rev. Sci. Instrum.* **69**, 3902 (1998).
4. S. Kalinin, A. Gruverman, *Scanning Probe Microscopy: Electrical and Electromechanical Phenomena at the Nano-Scale*, (New York: Springer: 2007).
5. B.I. Kharisov, O.V. Kharisova, U. Ortiz-Mendez, *CRC Concise Encyclopedia of Nanotechnology*, (CRC Press, Boca Raton: 2016).
6. <http://www.kelvinprobe.info/technique-principles.htm>
7. <http://www.kelvinprobe.info/technique-theory.htm>
8. A. Hossain, *Thesis: Design and Development of Home Automation System Controlled through Short Message Service (SMS)*, (Dhaka University of Engineering and Technology, Gazipur: 2009).
9. R. Condit, D.W. Jones, *Microchip Technology Inc*, DS00907A, 1 (2004).
10. <https://www.elprocus.com/digital-to-analog-converter-dac-applications/>
11. A. Al Mortuza, F. Pervez, K. Hossain, S. Kumar Sen, N. Haque Mia, M.K. Basher, S. Alam, *Univ. J. Eng. Sci.* **5** No⁴, 57 (2017).
12. A. Del Campo, E. Arzt, *Chem. Rev.* **108**, 911 (2008).
13. S.B. Eadi, H.S. Song, H.D. Song, J. Oh, H.D. Lee, *Coatings* **9**, 349 (2019).

Реалізація системи зонду Кельвіна для обробки поверхні напівпровідникаC. Zegadi¹, Z. Lounis², A. Haichour¹, A. Hadj Kaddour², D. Ghaffor²¹ *Micro and Nanophysics Laboratory (LaMiN), National Polytechnic School of Oran, ENP Oran-Maurice AUDIN, BP1523 El-Mnaouer, 31000 Oran, Algeria*² *LABMAT, National Polytechnic School of Oran, ENP Oran Maurice AUDIN, Oran, Algeria*

Знання електричних властивостей матеріалів неминуче в поверхневих технологіях, таких як мікро-технологія, корозія тощо. Що стосується поверхневих явищ, то робота виходу являє собою основну властивість. Вона була розроблена лордом Кельвіном і відповідає контактній різниці потенціалів між двома поверхнями матеріалів. У роботі збирання даних системи зонду Кельвіна (KPS) проводилося після послідовних випробувань в електронних обчисленнях і фізичних полях з метою отримання роботи виходу провідникових і напівпровідникових матеріалів. Ця система виявила велике значення управління напруженою підтримки V_b для обчислення конденсатора, застосованого в структурі метал-діелектрик-напівпровідник (MIS) для вимірювання поверхневого потенціалу напівпровідників. Деякі проблеми були вирішені під час створення системи, і відповідна частота 50 Гц була належним чином відрегульована. Однак вольт-амперне перетворення в KPS не проводилося через нечутливість використаних підсилювачів. Щоб зрозуміти цю складність експериментального дослідження сигналу, ми використали обчислення за допомогою коду Фортран. Воно підтвердило, що сигнал зонда Кельвіна має дуже слабку амплітуду порядку піковольта. З іншого боку, завдяки іншим доступним вимірювальним приладам, чутливість яких значно нижча, ніж сам сигнал, ці результати виправдовують експериментальні кроки.

Ключові слова: Контактна різниця потенціалів, Зонд Кельвіна, Структура MIS, Поверхневі явища.