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CONCEPTION OF THE KELVIN METHOD ON THE BASIS OF A MECHANIC-ELECTRICAL TRANSFORMATION

The Kelvin method was based on the concept of the dynamic capacitor recharging by a contact potential difference. The present paper draws attention to the fact that the contact potential difference is not the same physical agent as the electrical potential difference due to the electromotive force. It cannot act as an active electrical voltage and, accordingly, cause the flow of an electric recharging current. The real reason for the appearance of a measured signal is the transformation of the electrode movement mechanical energy into the electric current energy. The current is generated due to periodic changes in the screening conditions of electrostatic charges above the investigated surface. Investigations are made of the method sensitivity to the amount of charges on the sample surface. It is shown that the measurement results are interpreted without invoking the ideas of the work function. Therefore, the method can be successfully used in studies of organic and biological materials and electrolytes. The proposed mechanism is applicable in both the investigations of macroscopic distributions of the surface charge and the atomic scale in the Kelvin probe force microscopy.

Keywords: nondestructive testing, Kelvin method, contact potential difference, surface charge measurements.

1. Introduction

The Kelvin method is widely used for the nondestructive testing of the surfaces of materials. It is applicable to measure the work functions of solids [1]. The efficiency of the method in investigations of the charging of surfaces of dielectrics, semiconductors, and nanostructures was demonstrated in [2–4]. The results of investigation of conditions for the surface photo-emf occurrence and its magnitude measurements can be used for the improvement of solar cell characteristics [5]. The development of this method has led to the creation of Kelvin probe force microscopy [6]. It is considered that the measured signal in the Kelvin method is due to the action of a contact potential difference (CPD) [1, 7]. However, CPD is not equivalent to the electric voltage causing the current flow in an electric circuit. To obtain the current, it is necessary to apply an external force of a different nature than that of the electrostatic force in contacts. Heating or illuminating one of the contacts can cause such forces. Under the action of these forces, the Fermi energy gradient is established, and, accordingly, the directed motion of electrons is arisen (thermo and photo electricity). The only possible source of external forces in the Kelvin method can be associated with the reciprocating motion of

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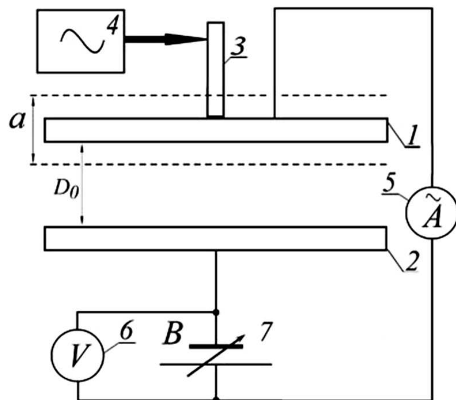

Fig. 1. Installation for CPD measurements with the use of a dynamic capacitor. *1* and *2* – plates of the dynamic capacitor, *3* and *4* – activator and AC source, *5* – null – indicator, *6* and *7* – voltmeter and DC source

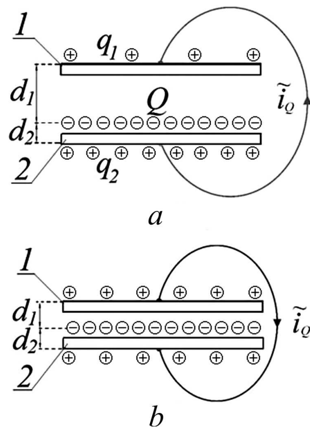

Fig. 2. Recharging the dynamic capacitor due to the motion of its plate

the dynamic capacitor plate. But this factor does not change the energy of electrons in a solid and cannot lead to the appearance of a Fermi energy gradient. To explain the reason for the appearance of a measured signal in the Kelvin method, a mechanism of mechanic-electrical transformations (MET) was proposed [8]. The consideration of the MET mechanism in the lecture course on "Electricity" will be useful for understanding the physical basic of the Kelvin method.

2. Theory of Measurements

The development of the Kelvin method became possible due to using the dynamic capacitor proposed in [9]. Experimental setups for applications of the method are being continuously improved [10, 11]. Basic components of a setup for CPD measurements between metals *1* and *2* are shown in Fig. 1.

An electrodynamic or piezoelectric vibrator activates the reciprocating motion of plate *1*. It is assumed that, in the circuit containing such a dynamic capacitor, the current appears [1, 7]:

$$\tilde{i}_c = \frac{dC}{dt}(U_{\rm DC} - U_{\rm CPD}) = \tilde{i}_{\rm B} - \tilde{i}_{\rm CPD}. \quad (1)$$

By regulating the value and polarity of the voltage $V_{\rm DC}$, one reduces this current to zero: $\tilde{i}_C = 0$. This is achieved, when $V_{\rm DC} = V_{\rm CPD}$. So, the measurements of $V_{\rm CPD}$ are based on the compensation principle. In compensatory methods, the real acting factors of comparison are not the voltages, but the electric currents caused by them. The null-indicator $\tilde{A}$ (Fig. 1) is destined for measuring these currents and for indicating the state of their mutual compensation: $\tilde{i}_{\rm CPD} = \tilde{i}_{\rm B}$. The current $\tilde{i}_{\rm B}$ is caused by recharging the capacitor by the voltage $V_{\rm DC}$ from the electromotive force of battery B. For the capacity of a unit area, we get

$$\tilde{i}_c = \frac{dC}{dt}U_{\rm DC} = \frac{\varepsilon_0 a\omega\cos\omega t}{\tilde{D}^2}U_{\rm DC}, \quad (2)$$

where ε_0 is the permittivity constant, a and ω are the amplitude and frequency of oscillations of plate *1*, and $\tilde{D} = D_0 + a\sin\omega t$.

The similar reason is generally assumed for arising the current $\tilde{i}_{\rm CPD} = \frac{dC}{dt}U_{\rm CPD}$. However, $U_{\rm CPD}$ cannot recharge the dynamic capacitor and be the direct cause for the current $\tilde{i}_{\rm CPD}$. In the electric circuit shown in Fig. 1, all contacts have the same temperature. Therefore, there is no the emf of contact nature. Recharging the dynamic capacitor and the generation of an alternating signal occur according to the MET mechanism [8]. We explain this mechanism, by using Fig. 2.

In the simplest case, we assume that plates *1* and *2* are made of the same metal. So, the contact potential difference between them does not arise ($U_{\rm CPD} = 0$). The layer of charges with density Q is located at distances d_1 and d_2 from the plates (hereafter, indices *1* and *2* denote the corresponding capacitor plates). The electric field of this charge induces the screening charges q_1 and q_2 on the capacitor plates (the charges' densities are taken per unit

surface). When plate *1* moves away from plate *2*, the ratio d_1/d_2 increases. This leads to a redistribution of the screening charges: q_1 decreases, and q_2 increases (Fig. 2, *a*). This causes the electric current to flow in the external circuit of the dynamic capacitor. Then plate *1* moves toward to plate *2* and the relations d_1/d_2, q_1/q_2 and current flow direction are changed in the reverse order (Fig. 2, *b*). The expression for the alternating current $\tilde{i_Q}$ arising in this process was obtained in [8]:

$$\tilde{i_Q} = \frac{dq_1}{dt} = \frac{d_2 Q\, a\, \omega \cos \omega t}{(\tilde{d_1} + d_2)^2}, \qquad (3)$$

where $\tilde{d} = d_1 + a \sin \omega t$.

The current $\tilde{i_Q}$ has a real electric power and can be measured by an external device $\tilde{A}$. In this technique, the dynamic capacitor is a mechanic-electrical transformer (MET). The electric field of the static charge becomes the source of the emf under the action of external forces caused by the mechanical reciprocating motion. The voltage measured by the voltmeter V, when $\tilde{A} = 0$, is the compensation voltage $U_{\rm CB}$. Its theoretical value can be found by equating the currents from formulas (2) and (3): $\tilde{i_{\rm B}} = \tilde{i_Q}$. Taking into account that $\tilde{D} = \tilde{d_1} + d_2$, we get

$$U_{\rm CB} = \frac{Q}{\varepsilon_0} d_2. \qquad (4)$$

Thus, by the Kelvin method, one can determine the properties of a charged layer situated above the investigated surface.

3. Results of Experimental Measurements

3.1. The charge on a dielectric layer

The verification of formula (4) was performed in model experiment. The measurements were carried out on a silicon sample with a layer of thermal oxide. The corona charging method was used to deposit a charge Q on the sample surface [12, 13]. The oxide thickness of 25 nm served as the d_2 gap in formula (4). The charge Q has been step by step incremented by adding the identical doses controlled by the time of deposition. Compensation voltage values $U_{\rm CB}$ were measured after each increment of the charge. The obtained dependence of $U_{\rm CB}$ on the number of doses n is shown in Fig. 3.

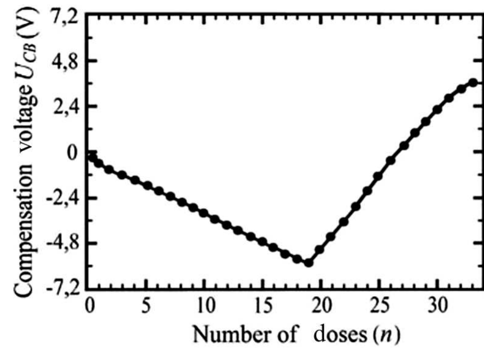

Fig. 3. Dependence of the compensation voltage $U_{\rm CB}$ on the number of doses n of the deposited charge

In the figure, two parts with opposite inclinations are seen at the $U_{\rm CB}(n)$ dependence: the LHS one was obtained by incrementing the positive charge density Q, whereas the RHS segment – under the reverse sign of the deposited charge. If a single deposition dose was Q_d, the charge $Q(n) = nQ_d$ corresponds to each n. Taking this into account, we see that the data of Fig. 3 show the $U_{\rm CB}(Q)$ dependence. It is seen that this dependence is a linear one. Hence, the mechanic-electrical transformations are realized in the experiments under the conditions that meet formula (4). Similar results were obtained on samples with SiO_2 layers over a wide range of thicknesses [8, 14]. Using formula (4), it is possible to determine the value of the charge density Q_d for a known layer thickness d_2. For example, from the data presented in Fig. 3, we obtain that Q_d values are 2.6×10^{11} and 6.7×10^{11} for positive and negative elementary charges per cm^2, respectively (here, the dielectric constant of SiO_2 is taken $\varepsilon = 4$). On the other hand, with the known density of the deposited charge, it is possible to determine the thickness of the dielectric layer. The charge deposition can also be used for the decoration of thickness variations in dielectric films. Heterogeneous oxide films cover the surfaces of silicon plates after their chemical cleaning. Measuring the $U_{\rm CB}$ distribution over the Si wafer surface is applied for a nondestructive control over the chemical processing quality [2, 3]. The *UV* pre-irradiation of the plate can be also used for the decoration of island films [15]. The possibility of detecting the charge covers has found its application in studying the adsorption-desorption processes [16]. Results of such research works can be used in the development of various sensors [17].

3.2. The charge forming the surface potential barrier

Above the atomically clean surface, there is a layer of negative charges, which prevents electrons to escape from a solid [1, 5]. This charged layer participates in the formation of a surface potential barrier. Its height is characterized by the work A, which must be done for an electron to escape into the vacuum. As any work, a product of the applied force f and the traversed path S determines it. The charge layer position above an atomically clean surface is schematically the same as near plate *2* in Fig. 2. Using the notation of parameters, as in this figure, we get

$$A = e\frac{q_2}{\varepsilon_0}d_2, \tag{5}$$

where eq_2/ε_0 is the electrostatic deceleration force f acting on the electron by the charged layer along the path d_2. In metals, the work A is equal to the work function W. Therefore, formula (5) can be used to estimate the value of the surface electron density n_l in the layer above an clean surface. Let us take the work function $W \approx 4.5$ eV. It corresponds to the value obtained in the emission studies of a large number of solids [1, 18]. The distance d_2 from the layer to the surface is unknown. Suppose that its minimum value is comparable to the interatomic distance of the crystal lattice ($d_2 \approx 1$ nm), and the maximum value exceeds it by the order of magnitude ($d_2 \approx 10$ nm). Being calculated for these two values of d_2, the magnitudes of n_l are 2.5×10^{13} cm^{-2} and 2.5×10^{12} cm^{-2}, respectively. Electrons move freely, and, because of the mutual repulsion, they are uniformly distributed in the plane of the layer. This distribution can be represented as a dynamically changing grid, which is not modulated by the crystal lattice. The period of such a grid corresponds to the interelectron spacing $\bar{l}$ averaged over the area. Calculations of the values of $\bar{l}$ at the maximum and minimum values of n_l given above showed that the electron lattice period is by 5 to 10 times larger than the interatomic distance in the crystal. The Kelvin method has been used only to determine the difference in the work function of plates of the dynamic capacitor ($eU_{\rm CB} = W_2 - W_1$). Therefore, it is necessary to consider a system, in which there are layers of charges above each of the plates. With oscillations of one plate, the mutual positions of all components of the system undergo periodic variations. Therefore, a charged layer above each plate will lead to the occurrence of charge exchange currents due the MET mechanism. Taking formula (6) into account, we obtain the compensation voltage for this case:

$$U_{\rm CB} = \frac{en_{l1}d_{21}}{\varepsilon_0} - \frac{en_{l2}d_{22}}{\varepsilon_0}, \tag{6}$$

where n_{l1}, d_{21} and n_{l2}, d_{22} are the charge densities and their distances to the surfaces of plates *1* and *2*, respectively. Thus, the measured value $U_{\rm CB}$ characterizes the difference in the heights of the surface potential barriers, and not the electrical potential difference, which can cause the Ohmic current flow. When studying the atomically clean surfaces of solids, the measured signal does not usually exceed 0.3 V [9, 20]. Assuming $d_{21} \approx d_{22} \approx 5$ nm, it follows from the calculation by formula (6) that, to obtain the signal $U_{\rm CB} \approx 0.3$ V, it is necessary that $n_{l1} - n_{l2} \approx 3 \times 10^{11}$ e/cm^2. In this case, n_{l1} only by 6 percents exceeds n_{l2}. The method enables the detection of even smaller differences in n_l on the investigated and reference surfaces, since the sensitivity of experimental installations usually is not less than 0.01 V [7, 10, 11, 13].

3.3. Dipoles on solid surfaces

A layer of dipoles above the solid surface can be the cause for the existence of a potential barrier that determines the value of work function [5]. PD inside the layer of N dipoles is the same as in a plane-parallel capacitor with a gap equal to the dipole length l:

$$U_{\rm C} = \frac{eN}{\varepsilon_0}l. \tag{7}$$

This potential difference prevents the escape of electrons from the solid, and, therefore, it affects the value of W determined by emission methods. As shown in [8], the same layer of dipoles, being investigated by the Kelvin method, determines the measured compensation voltage as

$$U_{\rm CB} = \frac{eNl}{\varepsilon_0}\frac{\ln 2d_2/l}{\ln 2d_1/l + \ln 2d_2/l}. \tag{8}$$

In this formula, the first term is the same as in formula (7). The second one may take values between 0.2 and 0.04, if we accept the realistic values of $l = 10^{-9}$ m and $d_1 = 3 \times 10^{-4}$ m, and d_2 is taken

between 10^{-9} m and 10^{-8} m. Thus, the dipole component in the surface barrier exerts a significantly weaker influence on the results of the work function measurements conducted by the Kelvin method, as compared to the emission methods. A simple explanation of this fact is that the MET-generated signal in a dynamic capacitor is caused by the external field of a dipole, which is essentially weaker than dipole's internal field influencing the emission currents.

3.4. Real surface of a solid

The real surface is one that is obtained after the mechanical treatment and chemical cleaning. It is covered with impurities and various chemical compounds (including oxide layers) in such degree that it is difficult to establish the physical boundary of a solid. Therefore, a simple single-layer model of the surface (Fig. 2) is not applicable. The signal in the circuit of a dynamic capacitor is formed by the overall action of all charges situated above the real surface. These include ions and polarized molecules on the surface and in an ambient gas. The charges may be accumulated, for example, on Si wafers in the process of chemical treatments used in microelectronics. In practice, the high sensitivity of the Kelvin method to a summary presence of charged objects is used.

3.5. Mapping electrostatic charge coverage over the sample surface

In most of the experimental setups, the reference electrode (plate *1* in Fig. 1) is made as a point probe. Usually, it has the flat end of a rod with a diameter of 0.2–0.5 mm. The investigated sample is scanned under the probe for getting the distribution of $U_{\rm CB}$ over the sample surface. The spatial resolution of the inhomogeneity in such installations is of the same order as the diameter of the reference electrode. The Kelvin method is called classical in such an application. Combining the methods of Kelvin and atomic force microscopy (AFM) allows one to increase the spatial resolution to the nanometer-scale imaging [6, 21]. In the Kelvin Probe Force Microscopy (KPFM), the reference electrode is the tip of the AFM cantilever (Fig. 4).

In the installation, an autonomous vibrator such as *3* in Fig. 1 is absent. The tip is brought into the reciprocating motion by applying the alternating voltage $V_{\rm AC}\sin(\omega t)$ between the cantilever and the sample. The voltage periodically changes the charge of the tip-sample capacitor. This leads to changing the attractive force between the elements of the capacitor ($F_C \sim CV^2$) and to the cantilever oscillation. There are three components of the force F_C: $F_C = \bar{F} + F_\omega + F_{2\omega}$, where $\bar{F}$ is a constant force, F_ω and $F_{2\omega}$ are its first and second harmonics. It is assumed that the first harmonic F_ω is due to the action of the contact potential difference $V_{\rm CPD}$. In the presence of a constant voltage $V_{\rm DC}$ in the circuit Fig. 4, the force F_ω reads [6, 21]

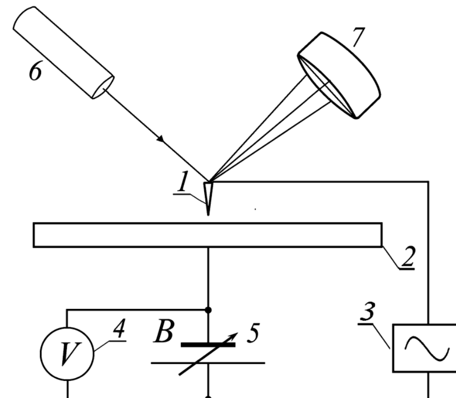

Fig. 4. Installation for Kelvin Probe Force Microscopy. *1* – tip of the AFM cantilever, *2* – sample, *3* – AC source, *4* and *5* – voltmeter and DC source, *6* – laser, *7* – position-sensitive detector

$$F_\omega = -\frac{\partial C(z)}{\partial z}(V_{\rm DC} - V_{\rm CPD})V_{\rm AC}\sin(\omega t). \quad (9)$$

This force can be nullified, if an applied external bias $V_{\rm DC}$ has the same magnitude as the $V_{\rm CPD}$, but with opposite sign. This condition has been detected by the absence of oscillations of the laser beam reflected from the cantilever at the frequency ω (Fig. 4). Comparison of formulas (1) and (9) shows that, in both classical Kelvin method and KPFM, it is assumed the same principle: $V_{\rm CPD}$ is compensated by $V_{\rm DC}$ ($V_{\rm DC} = V_{\rm CPD}$). But really, in the first method, the compensation of currents is used, whereas the second method employs the compensation of the force perturbations in the dynamic capacitor. $V_{\rm CPD}$ cannot be the cause for none of these factors, because it is not supported by any emf source. Just as in the classical Kelvin method, the results of KPFM studies are due to the MET mechanism. The first har-

monic F_ω is strongly affected by a short-range force between the tip and the charge situated above the sample surface. When the cantilever oscillates, the strength of the interaction is modulated because of the redistribution of screening charges, as shown in Fig. 2.

It is commonly accepted that the detected changes $U_{\rm CB}$, when scanning the samples, are due to variations in the work function at different points of the investigated surface. Such an approach can be applied, if the dimensions of the W inhomogeneity are at least by an order of magnitude greater than the surface interatomic distances. At the same time, KPFM allows one to detect the $U_{\rm CB}$ inhomogeneity on the atomic scale. The concepts of local work function and local contact potential difference (LCPD) are used in atomic-resolution KPFM [6]. Both of these concepts cannot correspond to those used in classical physics. Now, the LCPD characterizes the short-range force between the cantilever tip and the surface atoms. The detected $U_{\rm CB}$-inhomogeneities are due to the inhomogeneity in the charge density Q localized on the surface [formulas (4), (6), and (8)]. The work function and contact potential difference are not used in the MET concept. That is why it became possible to successfully apply KPFM for the investigation of surfaces of organic, biological materials [21–25], and even electrolytes [26]. The concept of the work function accepted for solids is not applicable to the mentioned materials at all.

4. Conclusions

1. The signal measured in the Kelvin method is due to the mechanism of mechanic-electrical energy transformations. In a dynamic capacitor, the mechanical energy of the movable electrode is transformed into the energy of the electric current. This current is generated due to periodic variations in the screening conditions of the electrostatic charge situated above the investigated surface. In the classical Kelvin method, the charging current of the dynamic capacitor is compensated, whereas, in KPFM, the force perturbations caused by this charge are suppressed.

2. The method makes it possible to determine the value of the deposited charge on the surface of the dielectric layer or its thickness. Estimates of the method sensitivity were carried out. The method makes it possible to detect the surface density of charges less than 10 percents of a monolayer. The high sensitivity of the method to the detection of charges underlies its technical applications. The method is employed for the nondestructive quality control over semiconductor wafers used in microelectronics. It finds its application to the study of adsorption processes, as well as in the development of the sensor technology and planar sources of photo-emf.

3. The results of the measurements are interpreted without using the concept of the work function. The Kelvin probe force measurements results show the charge distribution on the atomic scale. There is no need in the local work function, which has no real physical meaning. The method is applicable to the research of materials, to which the work function concept is not applied at all. It has found that the field of its application includes the study of charges on organic, biological materials, and electrolytes.

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КОНЦЕПЦІЯ МЕТОДУ КЕЛЬВІНА НА ОСНОВІ МЕХАНО-ЕЛЕКТРИЧНОГО ПЕРЕТВОРЕННЯ

Р е з ю м е

Застосування методу Кельвіна ґрунтується на концепції перезарядки динамічного конденсатора під дією прикладеної контактної різниці потенціалів. В даній роботі звертається увага на те, що контактна різниця потенціалів не є таким самим фізичним чинником, як і різниця потенціалів, що виникає під дією електрорушійної сили. Вона не може діяти як звичайна електрична напруга і, відповідно, викликати електричний струм перезарядки конденсатора. Реальною причиною появи вимірюваного сигналу є перетворення механічної енергії руху електрода в енергію електричного струму. Струм у динамічному конденсаторі виникає внаслідок періодичних змін умов екранування електростатичних зарядів, що розташовані над досліджуваною поверхнею. Проведені експериментальні дослідження вимірюваного сигналу методом Кельвіна в залежності від кількості заряду на поверхні зразка показали, що інтерпретація результатів вимірювань можлива без застосування поняття роботи виходу з твердого тіла. З цієї причини метод Кельвіна успішно використовується у дослідженнях матеріалів, до яких поняття роботи виходу не може бути застосовано взагалі: органічні та біологічні матеріали, а також електроліти. Запропонований механізм виникнення сигналу в методі Кельвіна слід враховувати при інтерпретації результатів досліджень як макроскопічних розподілів поверхневого заряду, так і в атомній силовій мікроскопії.