Principles of the Kelvin Probe Force Microscopy

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In this article a short review of the Kelvin Probe Force Microscopy (KPFM), the new technique for determining the work function based on the measurement of the contact potential difference is given. Brief historical development and the theoretical basis for the method are described. Some of the typical experimental results of KPFM are shown. Overview of one model and its results are discussed. Although the basic concepts of this technique are simple and can be described with the semi-classical model, KPFM gives results with the very good resolution.

I Introduction

Work function is one of the fundamental properties of materials. The precise definition of work function is based on a *gedanken* experiment in which an electron is removed from inside the bulk of a crystal and is transferred through the surface to the region outside, but not too far away from the surface. The distance of the electron from the crystal face should be large enough so that the image force can be neglected (typically 10^4 Å), but on the other hand it should be small compared with the distance from any other face of the crystal with a different work function[1]. The expression for the work function (at finite temperature), is given by:

$$eW = E_{vac} - \mu$$

where W is the work function, e is the electron charge, E_{vac} is the electrostatic energy outside of the bulk of the material described by the vacuum level and μ is the electrochemical potential of the electrons. Photoelectron emission spectroscopy can be used combined with scanning electron microscopy to obtain high spatial resolution maps of work function[2].

The Contact Potential Difference (CPD) for clean metal surfaces, which is proportional to the difference of the work functions of two materials ($V_{cpd} = \frac{\Delta W}{e}$; ΔW is the work function difference), is very sensitive to changes in the surface conditions (surface contamination, corrosion, oxides and oxide charges, changes in temperature, stress of crystalline structure, etc.). Therefore, obtaining information on CPD, one can have a better understanding of the physical and chemical properties of a material. Several researchers developed different techniques using a scanning tunnelling microscope to measure the local distribution of CPD on material surfaces[3–7]. These techniques have a resolution of the order of few nanometers, but are limited to conductive samples requiring a tunnelling current for operation. In this paper, we review the Kelvin Probe Force Microscopy- a technique which can be used for measuring CPD. It is the merging of two techniques: Kelvin Probe Method (KPM) and Atomic Force Microscopy (AFM); a brief overview of both techniques (KPM and AFM) is given bellow followed by a description, uses and limitations of the KPFM.

II Principles of the KPFM

In the Kelvin method for measuring CPD, which was first introduced by Lord Kelvin[8] in 1898 and further improved by Zisman[9] in 1932, two conductors are arranged as a parallel plate small spacing capacitor. A reference plate is vibrated (with frequency ω) close to the surface to be measured. Due to the periodic changes in distance between the plates, the capacitance varies with time, as does the charge on the condenser plates, which results in current flowing in the circuit that could be measured directly. Since the changes in the capacitance are also influenced by some ill-controlled factors, for the actual measurements of V_{cpd}, an adjustable dc voltage source V and a null-current detector are inserted in series in the circuit. The current in the circuit mentioned above is given by the following equation:

$$i\,(t) = (V - V_{cpd})\,\frac{dC}{dt}$$

where $\frac{dC}{dt}$ is the change of the capacitance in the interval of time dt. The voltage V is changed until the null-current condition in the circuit is detected by a sensitive current amplifier (technique of displacement

current). This compensating voltage is recorded as the contact potential difference between the two plates of the capacitor.

The AFM is based on the examination of the surface of a material involving a force type of interaction between an object and a fine tip. This gives a resolution about three orders of magnitude smaller than typical optical measurements. The first surface measurement using the attractive force microscope was performed by Martin *et al*[10]. A common arrangement for AFM consists of vibrating a tip at the resonance frequency of the metal wire using a transducer, where the wire acts as a cantilever. A laser heterodyne interferometer accurately measures the amplitude of the ac vibration. The gradient of the force between the tip and the sample modifies the compliance of the lever, hence inducing a change in the vibration amplitude due to the shift of the lever resonance. Knowing the lever characteristics ("spring constant", etc.), one can determine the vibration amplitude as a function of the tip-sample spacing in order to deduce the gradient of the force, and thus, obtain the force itself. The vibration amplitude of the lever also provides a feedback signal that allows the tip-sample spacing to be held constant.

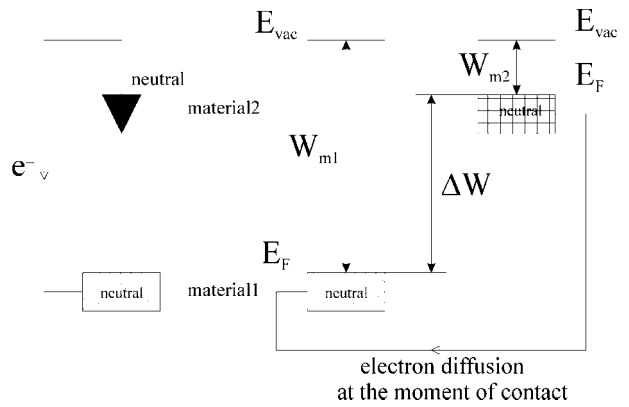
The principle of KPFM is the same as in the Kelvin method, except that forces are measured instead of current; this is explained as follows. Electrons in different materials have different chemical binding energy. When two different materials are electrically connected, electrons flow from the material with the smaller work function W_{m2} (weak binding) to the material with the higher work function W_{m1} (strong binding). This diffusion current builds up a double layer at the interface resulting in an electrostatic potential difference V_{cpd} between the two materials. This potential difference at the interface shifts the bulk electron energy levels until the Fermi level E_F of the two material is matched. When this equilibrium is reached, the electrostatic force cancels the diffusion force in the interface and the contact potential equals the difference of the two work functions.

Outside the bulk of the materials, the now different surface potentials generates an electrostatic force between the surfaces[11]. The electrostatic force between the tip and the sample is given by:

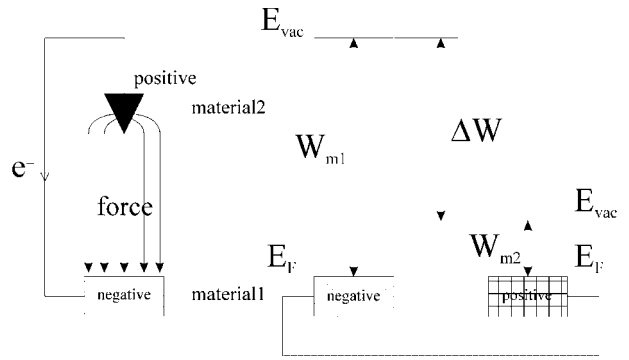
$$F_{es} = \frac{1}{2}\frac{dC}{dz}(V_{cpd})^2$$

where C is the capacitance between the tip and the sample surface and z is the distance between the tip and the sample.

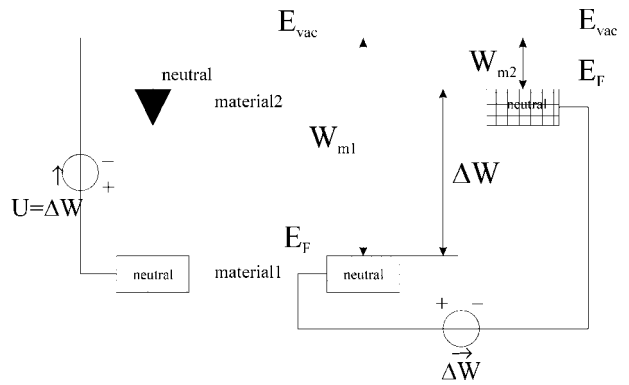
Similarly to the Kelvin Probe Method, in the KPFM method, direct detection of the amplitude of the vibration of the tip, which was assumed to be proportional to the force acting on the tip, depends on several ill-controlled factors, in particular $\frac{dC}{dz}$. Weaver and Abraham proposed[12] the so-called null-force method for detecting the potential between the detecting tip and the sample. An external voltage (the modulation ac voltage- $V_{ac} sin\omega t$ and the dc voltage- ϕ_{dc}) is supplied between the tip and the sample; the total potential can be written as:



a. Neutral tip and sample before being electrically connected.



b. Interaction force and electrostatic potential difference after electron diffusion.



c. External potential applied to zero the force.

Figura 1. Energy diagrams of a sample and the conducting tip (picture adapted from Jacobs *et al.*[17]).

$$V = V_{dc} - V_{ac}\sin\omega t$$

where V_{dc} is the total dc voltage ($V_{dc} = V_{cpd}$- ϕ_{dc}; V_{cpd}- surface potential, ϕ_{dc}- feedback voltage supplied to the sample surface). The electrostatic force can be written as:

$$F_{es} = \frac{1}{2}\left(\frac{dC}{dz}\right)\left(\left(V_{dc}^2 + \frac{V_{ac}^2}{2}\right) - 2V_{dc}V_{ac}\sin\omega t - \frac{V_{ac}^2}{2}\cos\omega t\right)$$

The spectral component of this force is proportional to V_{dc}^{18}. When the oscillation amplitude of the cantilever is zero, regardless of the capacitance between the tip and the sample, V_{dc} is also zero; therefore V_{cpd} is equal to ϕ_{dc}. In order to measure the surface potential, the dc level is changed until there is no detectable oscillation. If the voltage can be maintained at $V_{cpd}=\phi_{dc}$, the surface potential V_{cpd} can be obtained directly by measuring the external voltage ϕ_{dc}. The advantage of the null-force method for measuring CPD is that no calibration is needed since the null-force condition is detected and not the force itself.

III Theoretical basis of KPFM

The cantilever is a driven damped harmonic oscillator with an equation of motion given by:

$$mz'' + \left(\frac{m\omega_0}{Q}\right)z' + m\omega_0^2 z = F_0\cos(\omega_d t)$$

The cantilever steady state is given by:

$$z(t \leq 0) = A_0\cos(\omega_d t - \theta_0)$$

where

$$A_0 = \frac{\frac{F_0}{m}}{\sqrt{\left(\omega_0^2 - \omega_d^2\right)^2 + \left(\frac{\omega_0\omega_d}{Q}\right)^2}}$$

and

$$\theta_0 = \tan^{-1}\left(\frac{\omega_0\omega_d}{Q\left(\omega_0^2 - \omega_d^2\right)}\right)$$

ω_d is the frequency and F_0 is the amplitude of the driving force.

If we consider the case of an instantaneous step in $\frac{dF}{dz}$ at the time t=0, this results in an instantaneous shift in the resonant frequency from ω_0 to ω_0', changing the state of the cantilever to:

$$z(t \geq 0) = A(t)\cos(\omega_d t - \theta'(t))$$

where *A(t)* is the new amplitude and is given by:

$$\begin{aligned} A(t)^2 &= A_0'^2 + A_t^2 e^{-\frac{\omega'_0 t}{Q}} \\ &+ 2A_0'A_t' e^{-\frac{\omega'_0 t}{2Q}}\cos((\omega_t - \omega_d)t - (\theta_t - \theta_o')) \end{aligned}$$

The amplitude has three different components: the new steady-state term, a transient decay term, and a transient beat term. Only after a sufficient time will the vibration amplitude settle on a new steady-state value after a change in $\frac{dF}{dz}$ and ω_0.

The principle of ac detection involves detecting shifts in the resonance frequency of the cantilever which are caused by the interaction between the tip and the sample stray field. Thus, the method employed to measure the resonance frequency shifts of the cantilever becomes very important. For the *slope detection method*[10-12] the cantilever is driven at a frequency close to its resonance frequency, which is given by $\omega_0^2 = \frac{k_{eff}}{m}$. It is assumed that the force gradient ($\frac{dF}{dz}$) acting on the lever due to the interaction with the sample does not significantly affect the vibration mode of the lever, so that the effective spring constant can be written as $k_{eff} = k_L + \frac{dF}{dz}$, where k_L is the spring constant of the lever. Though the material and the geometry of the cantilever can be designed according to the needs of the experiment, thus changing the spring constant value, the first resonant frequency of the cantilevers is of order of 100kHz. A change in $\frac{dF}{dz}$ gives rise to a shift in the resonant frequency $\Delta\omega$, and a corresponding shift ΔA in the amplitude of the cantilever vibration. The frequency shifts are derived indirectly from the cantilever vibrating amplitude using an amplitude detector such as a lock-in amplifier. The bandwidth of the amplitude detector must be made sufficiently narrow to produce clear images, since the signal-to-noise ratio of the amplitude detector output is inversely proportional to its bandwidth. Studies have shown[3,11,13] that the minimum detectable force gradient is given by:

$$\delta F'_{\min} = \sqrt{\frac{2k_L k_B T B}{\omega_0 Q < z_{osc}^2 >}}$$

where $< z_{osc} >$ is the mean-square amplitude of the driven cantilever vibration, B is the measured bandwidth, Q is the quality factor of the resonance, and $k_B T$ is the thermal energy at the ambient temperature. It is possible to achieve very high Q values (10^4-10^5) by careful design and reduction of air damping in vacuum ($<10^{-3}$ Torr). For the slope detection method, however, increasing Q restricts the bandwidth of the system. Moreover if vacuum conditions are needed for some other reasons (e.g. to prevent sample contamination), it may not be possible to obtain low enough Q for an acceptable bandwidth and dynamic range. The response of the system may be expressed in terms of a

time constant $t=\frac{2Q}{\omega_0}$, which, for high Q values, gives a measurement time too long for most applications.

Albrecht et al.[14] used a *frequency modulation* (FM) *technique* for application of KPFM in vacuum condition. The cantilever serves as the frequency-determining element of the constant amplitude oscillator. The cantilever movement is sensed by a beam deflection. The beam from the laser diode is focused on the cantilever surface. The reflected beam is directed to the position sensitive photo-detector. The output of the detector is magnified by the feedback amplifier and positively fed back to the cantilever exciter piezo-device, thus forming an oscillator circuit with the cantilever as a mechanical oscillator. This loop circuit oscillates at the cantilever's resonance frequency if the loop gain is sufficient. The oscillation frequency is detected using a phase-locked loop (PLL) type frequency discriminator composed by a universal PLL integrated circuit. It can be shown[14] that the minimum detectable force gradient has an almost identical expression as for the slope detection method, but contrary to the later, in FM technique Q and B are independent. In the FM detection method, the Q depends only on the damping of the cantilever, and B is set only by the characteristics of the FM demodulator and therefore can be tailored for different applications.

IV Resolution and applications of the KPFM

An ac voltage ($V_{ac} sin\omega t$) is applied between the tip and the sample for most of the measurements of V_{cpd} with the force microscope. Since the amplitude of the vibration depends on the tip-surface spacing, one can use this signal to maintain the tip at a given distance from the surface. The feedback signal applied to the piezo-electric transducer of the lever moves the tip in the direction normal to the surface in order to follow the profile of the surface. The signal change (first resonance frequency) of the vibration (while the tip is approaching the sample), which is induced by the piezo-device, is used to control the tip-sample distance. The second feedback loop is used to measure the CPD by minimization of the electric field between the tip and the sample. The resonance frequency shift also occurs at the second resonance if the cantilever tip detects the force gradient; however its sensitivity is far lower than that of the first resonance. Thus the first resonance is normally used for tip height control and the second resonance is normally used for potential measurement. It can be shown[15] that the first resonance is about seven times more sensitive in detecting the force gradient for a cantilever having uniform cross section and mass per unit length along its whole length than the second resonance.

If the applied frequency of the ac voltage is the resonance frequency of the force sensor, the electrostatically induced amplitude A at ω is[12]:

$$A = \pi\varepsilon_0 V_{ac} V_{cpd} \left(\frac{QR}{k_L d}\right)$$

where R is the tip radius, d is the spacing between tip and sample and k_L is the spring constant.

The thermally induced amplitude noise N is given by:

$$N = \sqrt{\frac{2k_B TQB}{\pi k_L \omega}}$$

and therefore the sensitivity of the smallest measurable contact potential difference $V_{cpd,min}$ is given by:

$$V_{cpd,\min} = \sqrt{\frac{2k_B Tk_L B}{\pi^3 Q\omega}} \left(\frac{1}{\varepsilon_0 V_{ac}}\right) \left(\frac{d}{R}\right)$$

The noise in the z direction of the topographic image is comparable to the height of a single mono-layer. The KPFM technique gives a lateral resolution of the topographic image (defined by the width at half of the maximum of the derivative of the contact potential versus distance) of few tens of nm (even a resolution of a topographic image of 10nm can be achieved[16]). The sensitivity of the CPD which can be obtained by the KPFM method is in the mV range[17].

Although the KPFM technique has been developed few years ago (1991[20]), there is still some confusion regarding the correct name for this method. Some authors have adopted the name KPFM just when referring to experiments performed under UHV conditions; there are several other names for measurements performed at higher pressures: Scanning Potential Microscopy, Volta Potential Microscopy, Surface Potential Microscopy, Scanning Electrostatic Potential Microscopy. KPFM can be also regarded as a part of the more general Electric Force Microscopy.

The full potential of the KPFM method is still being determined; experimental measurements of the work function (at the eV level), based on CPD techniques, found in the literature, show a wide variation in values (few hundreds of mV[21,22]); the quantitative interpretation of the measured data obtained by the KPFM method is therefore difficult to be determined. Where measurements exist (for example for metals and semiconductors), the results obtained with KPFM are similar to reference values obtained by some other techniques[17,20,23].

Studies using KPFM can be divided into two main groups, according to the sample material being analysed: organic and inorganic materials. It follows below a brief discussion of those two areas of applications.

There is a growing interest on organic materials, such as polymers, oligomers, and liquid crystals, since these materials are easy to process and may be designed to present the desired physical or chemical properties. Analysis and characterisation of those materials

are therefore very important. However electrical conductivity is not a common property of organic materials; in fact, most organic materials are very poor conductors of electricity. Scanning Tunnelling Microscopy, which requires a tunnelling current for operation, is limited just to conductive samples, such as conductive polymers or doped polymers. Since KPFM is not limited by the electrical conductivity of the sample, valuable information on some properties of organic systems, otherwise hardly obtainable, can be gained. Apart from the fibrillar or grain structure and film formation, which can also be measured with some other Scanning Probe Microscopy techniques, KPFM can be used for the measurement of: local structural homogeneity of biomaterials, surface chemistry (i.e. surface tension), phase transition behaviour, properties of membranes in a physiological environment, etc. A good review of the use of Scanning Probe Microscopy for organic materials can be found in Ref.24.

There are two mains areas of applications of KPFM for inorganic materials: semiconductors and metal surfaces. A short description of each application is given below. The KPFM technique can also be used for material characterisation for nonotube applications[25].

Studies using KPFM for semiconductors are usually for work function variations due to the various doping materials (see for example Ref.26 and Ref.27) or with the measurement of the potential difference across Schottky junction (see for example Ref.28). Some other properties of semiconductor materials can also be obtained such as: the minority-carrier diffusion length[29], defects and discontinuities of the films[30] and others. A good review of applications of Scanning Probe Microscopy for integrated circuits is given in Ref. 31.

Two properties of metal surfaces, usually studied with KPFM technique, are: crystalline structures and changes of the surfaces induced by adsorbate materials. Since the value of work function depends on the crystalline facet, each grain with a certain orientation within the polycrystalline structure can be in principle detected. The phase transition, often followed by structure changes, and the changes between the crystalline and the amorphous structures can be observed using in situ KPFM. Since the work function is the surface property presenting one of the highest sensitivities for any surface contamination (either induced by adsorption or implantation), KPFM is often used in studies for metal corrosion or metal oxidation. A good review of the application of KPFM for molecular surfaces is given in the Ref.32.

V Modeling of the KPFM

Lateral resolution of the topographic image is influenced by the long-distance static electron force. Force microscopes operated using the KPFM method require a rather large ac modulation voltage between the sample and the tip in order to ensure an adequate sensitivity to variations in the contact potential (typically several volts modulation for one mV sensitivity). These high voltages will generate high electric fields (10^9 Vm^{-1} typically for 10V modulation and sample-tip distance of 10nm), which would probably affect the physical conditions at the surface of a specimen. In order to prevent cross-talk between topography and contact potential difference measurements a *lift-mode technique*[17] was developed. The specimen was first scanned in the tapping mode to determine the surface topography in order to obtain a potential image. During the scanning no external voltage was applied to the tip in order to keep long-range electrostatic interactions small. Using the built-in lift-mode feature, each acquired scan line was immediately retraced at a set lift height from the sample with the same pixel density as for topography, but using a compensating voltage.

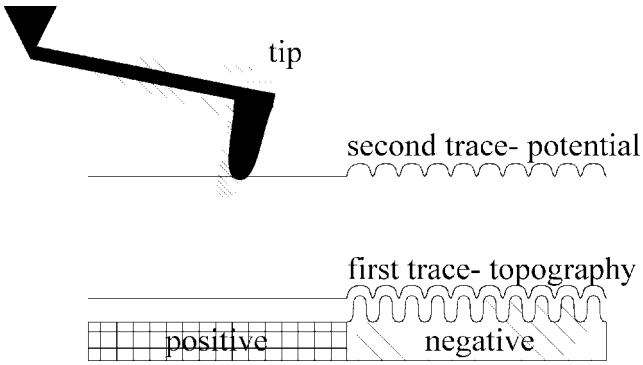


Figura 2. Topography is scanned in a first trace and immediately retraced at a set distance from the surface (lift height) while using feedback signal to measure surface potential. (picture adapted from Jacobs *et al.*[17])

Since the measurement of the surface potential depends on long-range electrostatic interactions between the tip and the sample, the resolution of the KPFM not only depends on the tip's apex, but also on the tip's side walls (though to a lesser extent). When the tip-sample distance is sufficiently small, the capacitance is determined mostly by the tip's apex. Usually a lift height of 10nm is normally sufficient for the cantilever to oscillate freely during a lift cycle. However, as the tip-sample distance increases above 100nm, the side wall area of the tip starts to govern the capacitance; the effective area of the capacitance, which determines the spatial resolution of a potential measurements, increases rapidly with tip-sample distance. Therefore, the role of the cantilever cannot be ignored in a quantitative analysis. In order to minimise the influence of the cantilever, it has been suggested[17] to modify the all-metal taping-mode cantilever holder by electrically insulating the leaf spring, holding the cantilever against the piezo, with a Teflon washer and nylon screw shielding the electrode against parasitic electric field and thereby improving the sensitivity and the lateral resolution of the probe. A thin wire was used for applying the necessary voltage.

Jacobs *et al.*[18] modelled the KPFM method assuming that the tip's apex is conductive and rotationally symmetrical. These researchers used a numerical

simulation method (multiple multipole program[19]) for deriving the contrast transfer characteristics of KPFM for:

1.small spots variable in their size

2. steps in the electric surface potential distribution

They modelled the KPFM set-up as a sample surface consisting of n areas with different surface potential ϕ_i and with the tip at the potential ϕ_t.

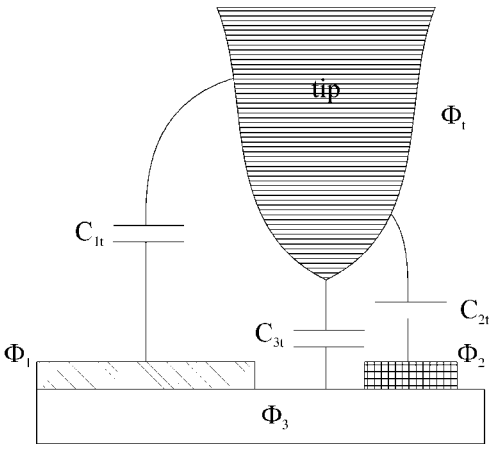


Figura 3. Model of KPFM set-up: System of ideal conductors with electrostatic interactions represented by mutual capacitances Cij. (picture adapted from Jacobs *et al.*[18]).

Using that approach, the electrostatic field energy is given by:

$$E_e = \frac{1}{2}\left(\sum_{i=1}^{n-1}\left(\sum_{j=i+1}^{n} C_{ij}\left(\phi_i - \phi_j\right)^2\right)\right)$$

$$+\frac{1}{2}\sum_{i=1}^{n} C_{it}\left(\phi_i - \phi_t\right)^2$$

The approximation C_{ij}=C_{ji} was used here, where Cit is the capacitance between the tip and the region. The electrostatic force is[18] is given by:

$$F_{es} = \frac{1}{2}\left(\sum_{i=1}^{n-1}\left(\sum_{j=i+1}^{n} C'_{ij}\left(\phi_i - \phi_j\right)^2\right)\right)$$

$$+\frac{1}{2}\sum_{i=1}^{n} C'_{it}\left(\phi_i - \phi_t\right)^2$$

where $C'_{ij} = \frac{dC_{ij}}{dt}$. The electrostatic force interaction between the tip and the sample surface depends both on the derivatives of the capacitances C_{ij} between the different regions on the surface and the derivatives of the capacitances C_{it} between the tip and the sample. It should be noted that when the tip is moved along the z axis towards the surface, the electrostatic coupling between different sample regions under the tip is disturbed and the corresponding coefficients C_{ij} decrease. Hence, the derivatives C'_{ij} are not zero and contribute to the force at the local tip location.

In the KPFM method an external ac voltage with adjustable dc offset is applied to the conductive tip, which gives the value: $\phi_t = \phi_{dc} + V_{ac} sin\omega t$. The first harmonic component of the tip force, depends only on the mutual capacitance between the tip and the surface C_{it}.

$$F_\omega = -\sum_{i=1}^{n-1} C'_{it}\left(\phi_i - \phi_{dc}\right) V_{ac}$$

The force component is measured and the feedback electronics adjust the dc potential offset ϕ_{dc} until F_ω vanishes. Setting $F_\omega = 0$, it is possible to obtain:

$$\phi_{dc} = \frac{\sum_{i=1}^{n-1} C'_{it}\phi_i}{\sum_{i=1}^{n-1} C'_{it}}$$

The measured KPFM potential ϕ_{dc} does not exactly match the surface potential bellow the tip, rather it is weighted average over all potentials ϕ_i on the surface, the derivatives of the capacitances, $C\ '_{it}$, being the weighting factors. The smaller and more distant a spot is the lesser contribution it gives. The potential of an isolated area will approach the value of the surrounding surface potential as the area decreases in size. Moving the tip across the potential step, the weighting factor C'it will increase, whereas all other weighting factors will decrease. Thus, for example, an ideal potential step on the surface will appear as a smoothed step gradually approaching the value ϕ_i.

The same authors[18] studied the total system response running simulations with different tip locations and different spot diameters. The optimum performance of KPFM is achieved when the sum of local electrostatic interactions is higher the sum of non-local ones. This ratio is favored by long and slender tips, provided that the tip apex is not too small (around 100nm).

VI Conclusion

Photo-electron spectroscopy coupled with scanning electron microscopy has been used for determining work function of surfaces for many years. The KPFM method might replace that technique, providing more reliable results, since its resolution, of the order of 1mV, is considerably higher than the resolution of the photoelectron method.

The KPFM method can be applied for determining CPD between surfaces in both vacuum and controlled atmosphere; different techniques (lift mode, slope detection, frequency modulation) have been employed for improving the accuracy of the method.

The KPFM method can be used as part of a controlling mechanism in ion implantation due to its high

sensitive and fast response; other possible areas of interest using the KPFM method include chemical and physical studies of clean and contaminated surfaces.

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