U.P.B. Sci. Bull., Series A, Vol. 78, Iss. 1, 2016 ISSN 1223-7027

DETERMINATION OF THE LOCAL DIELECTRIC CONSTANT OF INSULATING MATERIALS BY AN ATOMIC FORCE MICROSCOPY TECHNIQUE

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We report about quantitative measurements of the local dielectric constant of insulating materials. A conductive atomic force microscope (AFM) tip is biased relative to the sample and the resulting tip-sample electric force is measured as a function of the distance to the surface. Using a suitable analytical model, the geometric characteristics of the tip are determined from force measurements on conductive materials and are then used to calculate the local dielectric constant from the same type of measurements on insulating materials.

Keywords: local dielectric constant, atomic force microscopy.

1. Introduction

Quantitative determination of the local dielectric constant at the sub-micrometer scale is highly important, especially in the context of the continuously growing interest for small scale devices, from the field of micro-/ nano-electronics, to optics and biology. The dielectric constant influences properties such as the electronic states in quantum dots [1], the band gap in photonic crystals [2], the electric properties of nanocomposites [3,4].

The approach presented in this paper is based on modeling the AFM tip as a truncated cone with a spherical cap apex termination, in proximity of an infinite conductive surface [5]. From this model, an analytical formula for the tip-sample electric force was calculated. This is accomplished by dividing the effective surface of the tip into infinitesimal surfaces d*S* and calculating the electric force between each surface element d*S* and the underlying infinite conductive surface. The calculations are based on the assumption that the electric field at each infinitesimal surface element is the same as the electric field created by two infinite conductive planes having the same relative orientation. The contributions

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of the spherical cap apex and the truncated cone are integrated separately and summed at the end [5].

This model is represented in figure 1, where H is the total height of the tip, z is the distance between the apex and the surface of the sample and R is the radius of the apex; z_A, z_B and z_C are the distances from the surface to the large base and the small base of the truncated cone, and to the tip of the cone, respectively. The half-angle of the cone is θ. The tip is electrically biased relative to the conductive sample at a potential V.

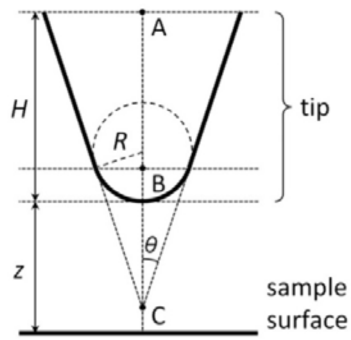


Fig. 1. The tip modeled as a truncated cone with a spherical cap apex, in the vicinity of the surface of the sample

The contribution of the apex to the total force is

$$F_{\text{apex}} = \pi\varepsilon_0 R^2 \frac{1-\sin\theta}{z[z+R(1-\sin\theta)]} V^2 .$$

The calculation for the contribution of the truncated cone gives

$$F_{\text{cone}} = \frac{\pi\varepsilon_0 V^2}{[\ln\tan(\theta/2)]^2}\left[\ln\frac{z_A}{z_B} + z_C\left(\frac{1}{z_A} - \frac{1}{z_B}\right)\right].$$

The calculation was made considering that $z_A >> z_B$ and $z_A >> z_C$. Next, by imposing a continuity condition between the apex and the truncated cone surfaces, the total force is calculated as the sum of the two contributions [5]:

$$F_{\text{total}}(z) = \pi\varepsilon_0 V^2\left[\frac{R^2(1-\sin\theta)}{z[z+R(1-\sin\theta)]} + \frac{1}{[\ln\tan(\theta/2)]^2}\left(\ln\frac{H}{z+R(1-\cos\theta)} - 1 + \frac{R(\cos\theta)^2}{\sin\theta[z+R(1-\sin\theta)]}\right)\right]. \quad (1)$$

Using the relation between the electric force and the vertical gradient of the tip-sample capacity, $\mathrm{F_{total}(z)} = -\frac{\partial \mathrm{C}}{\partial \mathrm{z}}\frac{\mathrm{V}^2}{2}$, equation (1) leads to

$$\frac{\partial C}{\partial z}(z) = 2\pi\varepsilon_0 \left[\frac{R^2(1-\sin\theta)}{z[z+R(1-\sin\theta)]} + \frac{1}{[\ln\tan(\theta/2)]^2}\left(\ln\frac{H}{z+R(1-\cos\theta)} - 1 + \frac{R(\cos\theta)^2}{\sin\theta[z+R(1-\sin\theta)]}\right)\right]. \quad (2)$$

These relations show that the electric force exerted on an electrically biased tip in the close proximity of a conductive surface can be described as a function of the geometric parameters of the tip (R, θ and H), the bias V, and the distance to the surface, z. This way, the geometric parameters can be obtained from force-distance measurements (F vs. z curves) on conductive surfaces, by fitting the results of such measurements with equation (1).

Next, the geometric parameters are employed in the calculation of the dielectric constant for insulating materials. A modified equation is used, which takes into account the presence of the dielectric material (equation (3)). This time, the geometric parameters are known and the value of the dielectric constant is obtained by fitting the measured values of the electric force (or capacitance gradient) with equation (3).

$$\frac{\partial C}{\partial z}(z) = 2\pi\varepsilon_0 \frac{\varepsilon-1}{\varepsilon+1}\left[\frac{R^2(1-\sin\theta)}{z[z+R(1-\sin\theta)]} + \frac{1}{[\ln\tan(\theta/2)]^2}\left(\ln\frac{H}{z+R(1-\cos\theta)} - 1 + \frac{R(\cos\theta)^2}{\sin\theta[z+R(1-\sin\theta)]}\right)\right] - -\left.\frac{\partial C}{\partial z}\right|_{z=z_{\mathrm{ref}}} \quad (3)$$

The steps described above are summarized in figure 2.

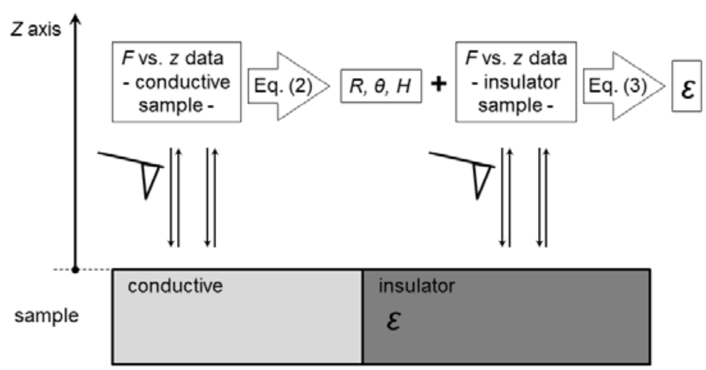


Fig. 2. Determination of the local dielectric constant

2. Experimental

The determination of the electric force acting on the AFM tip depends on the accurate measurement of the cantilever deflection and the calibration of the cantilever force constant k. We measured the cantilever deflection with the usual laser lever detection technique employed in common AFM experiments: a laser beam is reflected off the back of the cantilever and falls on a segmented position sensitive photodetector. The electric signals from the photodetector are used to determine the deflection of the cantilever with sub-nanometer accuracy. The experiments were carried out on two different AFM systems: a home-built system interfaced with commercial controllers (SPM1000 and PLLPro2 from RHK Technology) and a commercial AFM (XE100 from Park Systems). We used conductive coated cantilevers from Mikromasch with nominal force constants of 0.22 N/m. We determined the force constant using the approach described below.

Determination of the elastic constant of AFM cantilevers

The AFM cantilever is modeled as an elastic harmonic oscillator with one degree of freedom, corresponding to the vertical movement (deflection) of the AFM tip (Fig. 3). The damping caused by the fluid in which the cantilever oscillates (in this case atmospheric air) is proportional to the oscillation speed. The approach of Sader [6,7] gives a relation between the elastic constant of a rectangular cantilever and measurable quantities such as quality factor, its linear dimensions, and the mechanical characteristics of the fluid in which the oscillation takes place:

$$k = 0.1906\,\rho\, b^2\, L\, \Gamma_i(\omega_{res})\, \omega_{res}\, Q\,. \tag{4}$$

Here ρ is the density of the fluid surrounding the cantilever, b and L are the width and the length of the cantilever, respectively; $\Gamma_i(\omega_{res})$ is the imaginary part of the hydrodynamic function of the cantilever in the fluid, ω_{res} is the resonance frequency and Q the quality factor of the cantilever. The quality factor of an oscillating system is defined as the ratio between the stored energy and the energy dissipated in each oscillation cycle, at its resonant frequency.

$$Q = 2\pi \left.\frac{E_{stored}}{E_{dissipated}}\right|_{\omega=\omega_{res}}$$

For small oscillations of the cantilever the stored energy is proportional to the force constant and the square of the amplitude: $E_{stored} = ½\, k\, A^2$.

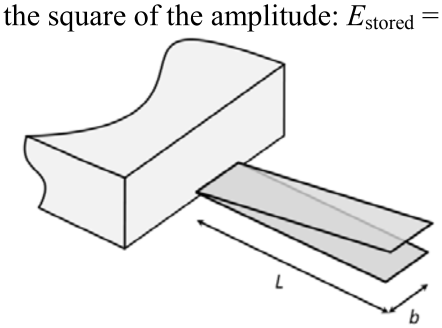


Fig. 3. The AFM cantilever modeled as a harmonic oscillator with one degree of freedom [6,7]

Relation (4) is valid for rectangular cantilevers with length to width ratio larger than three and Q value larger than unity, which was always the case for our studies.

The hydrodynamic function depends only on the Reynolds number (Re), defined as:

$$\mathrm{Re} = \frac{\rho L^2 \omega_{\mathrm{res}}}{\eta},$$

where η is the fluid viscosity surrounding the cantilever. The determination of the hydrodynamic function requires very complex calculations and an analytical solution has been given by Sader, in the form of a complex function which uses third degree Bessel functions [8].

The resonance frequency ω_{res} and the quality factor Q of the cantilever are accessible to direct measurements. The resonance peak is obtained from a frequency sweep, which records the oscillation amplitude as a function of the excitation frequency, for constant excitation amplitude (Figure 4). The quality factor can be calculated by the ratio between the resonance frequency, ω_{res}, and the value $\Delta\omega$, which represents the width of the peak where the amplitude decreases by a factor of $\sqrt{2}$:

$$Q = \frac{\omega_{\mathrm{res}}}{\Delta\omega}.$$

Using this model, the elastic constant of rectangular cantilevers can be calculated by measuring their plane dimensions b and L (usually by optical or scanning electron microscopy) and their resonance curve (usually by AFM).

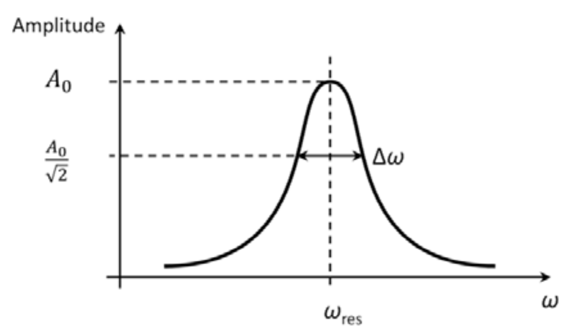
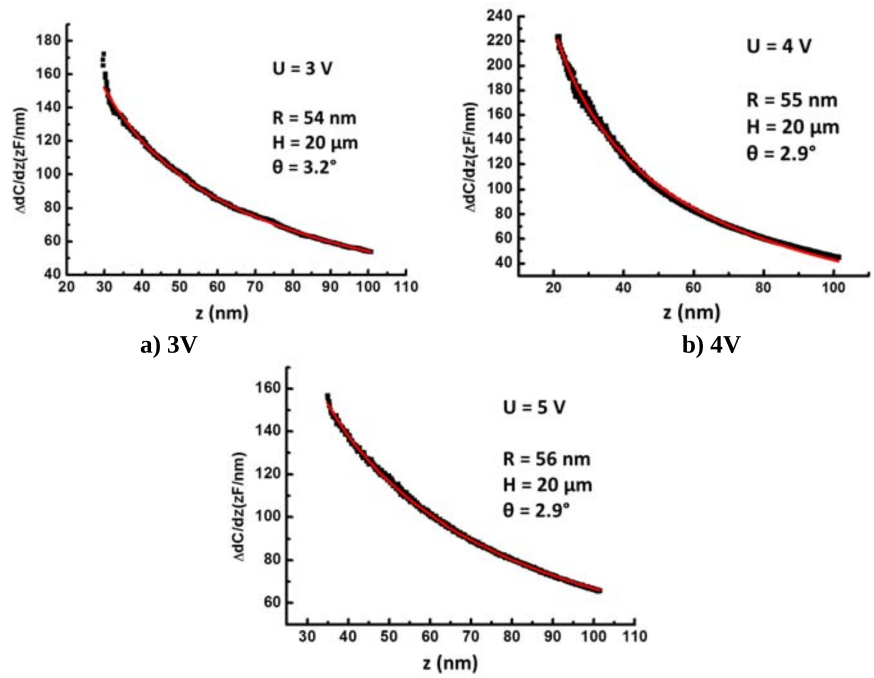


Fig. 4. The typical shape of the resonance peak for an AFM cantilever

By this method, our determinations gave the values of 0.26 N/m, 0.33 N/m and 0.24 N/m for the elastic constant of three cantilevers of the same type. These values are in good agreement with the nominal elastic constant specified by the producer, which was 0.22 N/m for the respective set of cantilevers.

Determination of the calibration curves on conductive surfaces

Force vs. distance calibration curves were recorded on conductive samples in order to determine the geometric parameters of the tip using equation (2). Examples of such curves are shown in Figure 5, for tip-sample bias voltages between 3 and 5 V and for measurements performed with the same tip, on a conductive (HOPG) substrate. As can be seen, the geometric parameters R, θ and H are well determined. Also, their values fit well with the nominal values specified by the producer for the AFM tip.



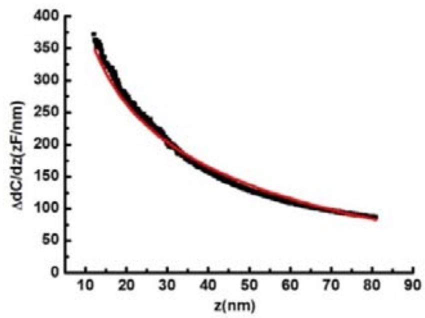
c) 5V

Fig. 5. Calibration curves for the same tip at different voltages, obtained from measurements on a HOPG surface

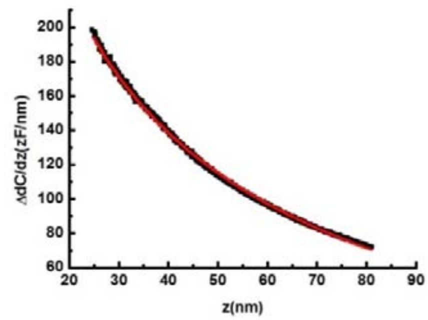
Determination of the dielectric constant for insulating materials

Next, the same kind of measurements were performed, on an insulating material (SiO_2) instead of conductive. The geometric parameters determined as described above were used with equation (3) to determine the dielectric constant of the insulating material. Data obtained from force vs. distance measurements above the SiO_2 surface were fitted with equation (3), in which the geometric parameters R, θ and H are known, and the calculated parameter is the dielectric constant ε.

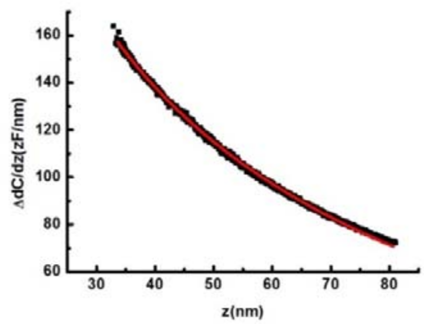
The results are shown in figure 6, for force measurements on a SiO_2 surface with tip-sample bias voltages between 3 and 5V. It can be seen that the determined values of the dielectric constant are in good agreement with values from literature (3.9) [9,10].



a) 3V, ε = 3.8



b) 4V, ε = 3.7



c) 5V, ε = 3.5

Fig. 6. Results of measurements on a SiO_2 surface, fitted with equation (3)

6. Conclusions

An analytical model for electric tip-sample interaction in AFM experiments is presented, which provides the basis for the quantitative determination of the local dielectric constant for insulating materials. In order to be able to accurately measure the electric force exerted on the tip, calibration measurements were carried out for the determination of the elastic constant of AFM cantilevers. The geometric parameters of AFM tips (height, curvature radius and cone angle) were determined from electric force measurements on conductive surfaces. Electric force measurements on SiO_2, fitted with the presented analytical model, provided the quantitative value of the local dielectric constant of the material.

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