APPLICATION NOTE 1

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The application note of the Marie Curie Skłodowska European Training Network

Wideband electrostatic force microscopy (EFM) for nanoscale dielectric spectroscopy

INTRODUCTION

A number of electrical atomic force microscopy (AFM) techniques are available that are capable of sensing the nanoscale electrical properties of samples ranging from new generation semiconductors over 2D materials to those of biophysical interest. Electrical AFM modes can be distinguished by current/impedance sensing and force sensing techniques as shown in Fig. 1, with each technique having their own advantage and operation frequency. Current and impedance sensing techniques (upper part in Figure 1), include techniques such as conductive AFM, scanning impedance microscopy and scanning microwave microscopy SMM. In them, the electrical signal (electric current or scattering parameters) coupled through the AFM cantilever is readout with an external electronic circuitry, for instance a current amplifier or a network analyzer, and they cover the range from DC (c-AFM) to 1-20 GHz (SMM). Force sensing techniques (lower part in Figure 1) rely on the optical readout of the cantilever deflection, like EFM (electrostatic force microscopy) and KPFM (Kelvin probe force microscopy). They are typically implemented to characterize static or low frequency electric properties and quasi-static dielectric properties up to roughly 100 kHz. With the emergence of short cantilevers, implementations up to few MHz have been reported (1).

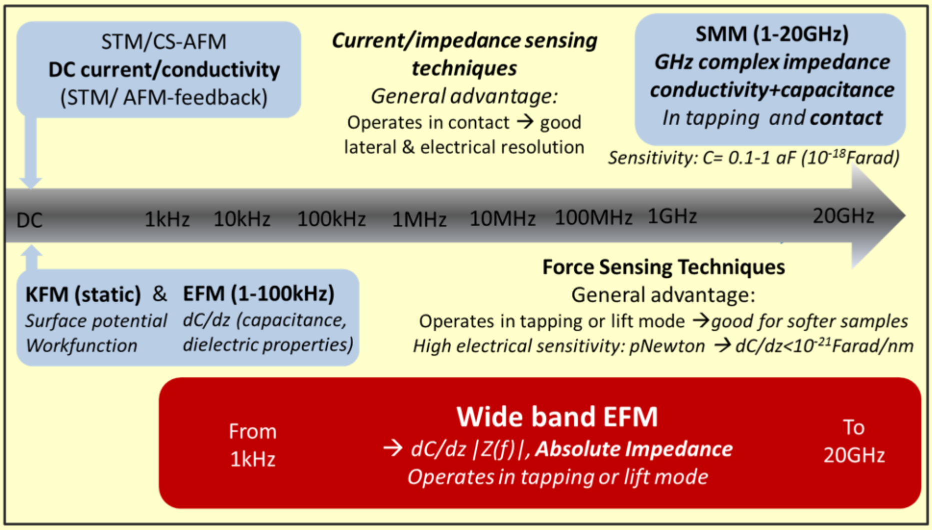

Figure 1. Electrical AFM techniques and their frequency range. SMM (scanning microwave microscopy); KFM (Kelvin force microscopy); EFM (electrostatic force microscopy); CS-AFM (current-sensing).

Here we describe the implementation of wideband EFM (described originally in Refs. 2,3, with further applications shown more recently in Refs. 4-6,7) that extends the available EFM frequency range from the kHz range to the GHz range, which allows for detection of the frequency dependent dielectric properties of samples. Compared to SMM, wideband EFM can work also below 1 GHz and has higher electrical sensitivity but only the magnitude of the force can be measured. While SMM can also work in contact mode, EFM is operated in tapping or lift mode which reduces the tip wear and sample modifications. Compared to standard EFM, the higher frequency range of wideband EFM allows exploring dielectric relaxation processes not accessible to conventional EFM.

APPLICATIONS

EFM addresses low to mid frequency dielectric properties of materials. Extending the frequency range in wideband EFM allows for detecting frequency dependent dielectric relaxation processes occurring at much higher frequencies. This versatile approach enables characterization of unique properties such as local dielectric spectrum in a wide frequency range, including conductive material properties (for instance, how carrier mobility of 2D materials changes with frequency). Additional insights such as, nanoscale spatial variation of electrical response at high frequencies of polymer blend structures and subsurface imaging of hard and soft materials with different electrical properties (e.g. silicon 3D devices) are expected to be possible. Furthermore, investigations of more complex biological systems in liquid environment based on the water dielectric constant and biomolecules hydration could be accessible.

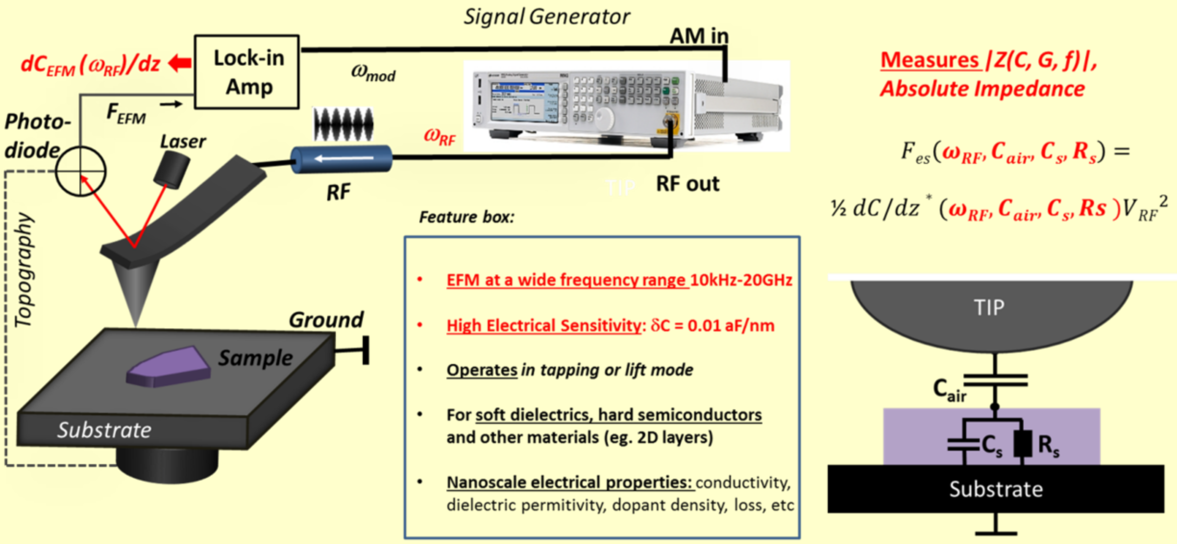

Figure 2. Wideband EFM setup. Left: The AFM is interfaced with a signal generator (Keysight MXG N5183B) which delivers an amplitude modulated signal to the conductive AFM probe. The tip holder of the AFM is designed to deliver electrical signals from DC up to 20 GHz to the probe, which is the same nose-cone used also for SMM. The cantilever starts to oscillate at the modulation frequency and detects the local complex capacitance of the sample as function of the carrier frequency. Right: Wideband EFM measures the electrostatic forces F_{es} at a given frequency ω_{RF} as a function of capacitance (C) and conductance (G). The circuitry model describes how F_{es} depends on the sample impedance.

OPERATIONAL PRINCIPLE

In conventional EFM the electrostatic force arising when a sinusoidal electric field is applied between a conductive probe and the substrate is measured. The electrostatic force consists of three components, the DC (DC-EFM), the first harmonic, ω (KPFM), and second harmonic, 2ω (AC-EFM). Detection of the force components is done by a lock-in amplifier. For AC-EFM only low frequencies (below the mechanical resonance of the cantilever) can be measured, typically in the kHz range. In wideband-EFM the down-converted DC signal is measured, and therefore the measurement can be done in a wide frequency range from kHz to GHz which is well above the mechanical resonance of the cantilever. To increase sensitivity and accuracy a heterodyne detection approach is applied based on the amplitude modulation of the high frequency signal (Fig. 2). A signal generator with a high frequency range delivers a carrier signal with a frequency ω_{RF}, which is modulated by amplitude modulation (AM) at a much lower frequency, ω_{mod}. Due to the modulation and to the down-converted DC signal, the cantilever starts to oscillate at the modulation frequency. This oscillation is detected by the AFM photodiode and the lock-in amplifier, giving information on the local complex capacitance gradient at ω_{RF}. The complex capacitance gradient dC*/dz depends also on the resistive/conductive properties of sample as detailed in Fig. 2. Selection of the carrier frequency ω_{RF} allows for estimation of capacitive and resistive properties of the sample under study up to the GHz range.

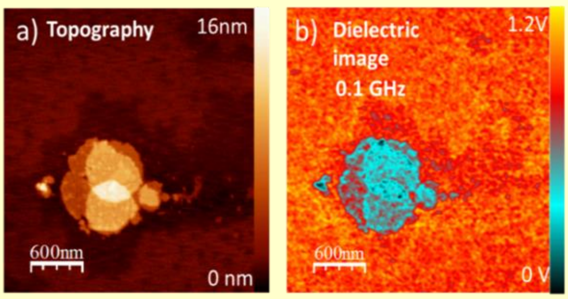

Figure 3. Dielectric measurement of purple membrane on mica (halobacterium salinarium). a) AFM Topography b) Wideband-EFM signal acquired in lift-mode (10nm lift-height) at f=100 MHz.

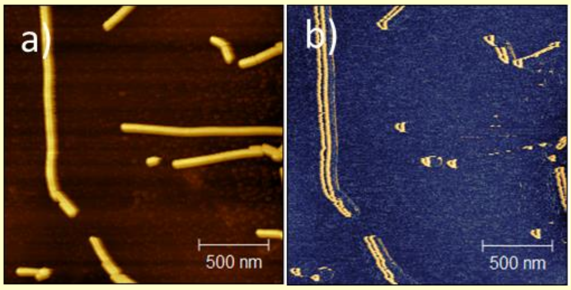

Figure 4. Wideband EFM mapping of tobacco mosaic virus on Si. A) AFM topography b) Electrostatic force response at 250 kHz.

A dielectric measurement of a biological systems such as membrane patch (halobacterium salinarium) is shown in Fig. 3. Single, double and triple layer of the membrane with a height of 5nm to 15nm are visible. The cell membrane which contains mainly protein has a dielectric constant of about 3 and is measured at a frequency of f=100 MHz. Additionally, by varying environmental parameters such as humidity and temperature characterization of dependent dipole relaxation is possible. Fig 4. Represents topography and electrostatic force response of tobacco mosaic virus (TMV) under 20% RH and 20 degrees Celsius

REFERENCES

1. M. J. Cadena et al (2016) ACS Nano **10**, 4062−4071
2. Hou A et al., Optical Electronics, **28** (1996) 819-841
3. Leyk A et al., IEEE MTT.S Digest (1996) 1525-1528
4. Gramse G et al., (2012) APL **101**, 213108
5. Umeda K. et al., (2012), APL **101**, 123112
6. M. Labardi et al. (2013) J. Non-Cryst. Solids **379**, 224
7. G. Gramse et al. (2019) Nanoscale **10**, 4303-4309