A new method for simultaneous Shear Force and Radiofrequency-Microwave analysis in Scanning Probe Microscopy: morphological, viscoelastic and dielectric properties of materials

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Introduction

We present a novel method that allows the simultaneous acquisition of the sample topography and viscoelastic and dielectric ε(ω) properties at the nanoscale. The low force of probe-to-sample interaction, characteristic of the shear force mode of operation, opens to the easy investigation of biological matter and more in general of soft samples minimizing the specimen deformation, damaging and dislodging. The shear force method also provides the viscoelastic properties of the sample surface and of thin liquid films nanoconfined between the probe apex and the underneath sample area. To this purpose we used a microcantilever set with its long axis perpendicular to the specimen surface (VOC, vertically oriented cantilever configuration). The morphology of the specimen is thus acquired using the shear force signal variation between the sample surface and the VOC apex that is excited to oscillate in a direction parallel to the sample surface. The probe is only sensitive to horizontal forces (shear forces) because of the high vertical spring constant of the cantilever.

Then, in order to acquire dielectric ε(ω) and RF-microwave material-related properties we have connected the VOC to a vector network analyzer, VNA (RF range 100 kHz–8.5 GHz) to measure RF impedance signal variations at the VOC apex-sample interface or through the specimen thickness [1, 2]. The VOC probe-to-sample configuration is very important because minimizes the parasitic capacitance of the cantilever.

Materials and Methods

A Multimode® atomic force microscope, AFM (Digital Instruments Inc., Santa Barbara, CA, USA), interfaced with a Nanonis AFM control system (Nanonis: SPECS Zurich GmbH, Zurich, Switzerland) equipped with two oscillation controller modules (with digitally integrated PLL/lock-in), was modified to perform shear force microscopy [1]. The vertically oriented cantilever, VOC, was connected to an Agilent ENA 5071C vector network analyzer (Agilent Technologies Inc., Santa Clara, CA, USA). To perform RF analysis, the microwave signal generated by the VNA is transmitted to the VOC and part of the signal is reflected (or transmitted) depending on the probe-to-sample impedance mismatch (or through specimen properties). The probe-to-sample mismatch variation is dependent on the material local properties of the sample and on the probe-to-sample distance at the nanoscale. For instance, the ratio of the generated microwave signal and the reflected one is measured by the VNA as a scattering reflection signal, S_{11}. The complex scattering parameter S_{11} is related to the probe-to-sample impedance by the following equation:

$$S_{11} = \frac{Z_L - Z_0}{Z_L + Z_0}$$

where Z_0 is the characteristic impedance of the transmission line, typically 50 Ω , and Z_L is the load impedance, which is equivalent to the impedance at the probe-to-sample interface. Through the VNA, we measured the variation of the modulus $|Z_L|$ and phase θ of the complex impedance $Z_L = |Z_L|e^{i\theta}$ at the probe-to-sample interface.

Results and Conclusions

1) Polystyrene spheres, muscovite crystallographic steps and DNA single molecules were used to test the performance of the shear force mode. The polystyrene spheres, with a mean diameter of 194 nm ± 5%, were used for the x–y calibration, whereas the stacking of 1 nm thick layers of the crystal structure of a commercial muscovite was used to test the z resolution of the system. The spatial resolution of the shear force mode in biomolecular imaging was assessed using as a test specimen DNA single molecules deposited on an atomic flat substrate. These observations demon-

strated the reliability of the shear force device and the low induced deformation, damaging and dislodging when dealing with biological matter and more in general with soft materials [1].

2) We used atomic flat graphite with the nanosized polystyrene spheres onto to test the simultaneous shear force and RF-microwave method. Impedance spectra were acquired in the frequency range from 100 kHz to 8.5 GHz [2,3].

We found a strong dependence of the RF impedance phase θ from both the probe-to-sample distance and the type of material (graphite or polystyrene). The RF impedance signal was highly sensitive to very short probe-to-sample distances (Figure 1), with an exponential trend that follows the empirical formula:

$$\theta(z) = a \cdot e^{-b \cdot z} + c$$

where z is the probe-to-sample distance, a, b and c are calibration parameters.

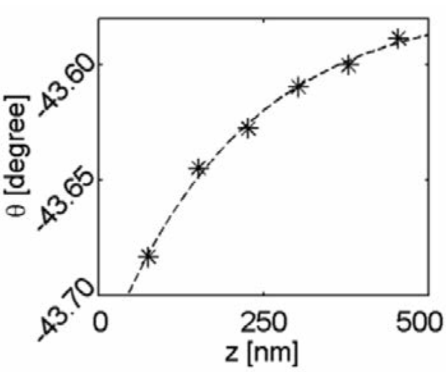

Figure 1. Exponential approximation (dashed line) of the experimental trend (stars) of the RF impedance phase signal versus the probe-to-sample separation z.

Through shear force and RF impedance variation analysis we performed a detailed characterization of the probe-to-sample shear nanocontact, where the sensing element for both the shear force and the radiofrequency signals is the apex at the free end of the VOC. The probe was characterized to behave like a capacitive non-contact probe during shear force observations, at least for frequency <100 MHz. Whereas, the impedance shows a highly variable signal characteristic of the type of cantilever and the experimental set-up at high frequency (>100 MHz).

In our configuration at a fixed RF, dynamic experiments of approach and withdrawal of the probe revealed a switch-like behavior related to a drastic change of the impedance from a non-contact/engage-contact regime to the contact regime which nullifies the free mechanical oscillation amplitude of the cantilever. Different types of cantilevers presented similar trends of the impedance modulus and phase as a function of the probe-to-sample distance, being the Δθ variation independent of the cantilever length and shape.

It is expected that the versatility of the presented Shear Force and RF-Microwave SPM-based method will be of great help to investigate the nanoscale morphology, viscoelastic and dielectric ε(ω) properties of a wide spectrum of materials, from metallic to dielectric and biological matter. Further work is ongoing to develop a quantitative methodology using calibrated standards.

References

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