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Determination of the dielectric constant of non-planar nanostructures and single nanoparticles by electrostatic force microscopy

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Abstract

Electrostatic Force Microscopy has been proven to be a precise and versatile tool to perform quantitative measurements of the dielectric constant of thin film domains in the nanometer range. However, it is difficult to measure non-planar nanostructures because topographic crosstalk significantly contributes to the measured signal. This topographic crosstalk due to distance changes between tip and substrate measuring non-planar surface structures is still an ongoing issue in literature and falsifies measurements of the dielectric constant of nanostructures and nanoparticles. Tip and substrate form a capacitor based on the contact potential difference between the tip and substrate material. An increase of the distance between tip and substrate causes a repulsive force while a decrease causes an attractive force. Thus, measuring in the so-called lift mode scanning the surface in a second scan following the topography determined by a first scan leads to a mirroring of the non-planar surface structure in the electrostatic signal superimposing the signal from dielectric contrast. In this work we demonstrate that the topographic crosstalk can be avoided by using the linear mode instead of the lift mode. The use of the linear mode now allows the determination of the dielectric constant of single nanoparticles.

1. Introduction

In recent years, Electrostatic Force Microscopy (EFM) has attracted more and more attention to determine dielectric constants of thin films [1–3], nanodimensional interfaces [4, 5], nanostructured systems [6] and nanoparticles [7]. The advantage of using EFM instead of conventional methods, such as ellipsometry, is the increased lateral spatial resolution, which opens the possibility to map dielectric constants in the nanometer range. Nevertheless, it is limited to very smooth surfaces as the surface induces topographic crosstalks, falsifying the obtained dielectric constants [8, 9]. The EFM (phase or frequency) signal often resembles the topography, as reported in various publications [1, 6, 10]. This effect is also observed in Magnetic Force Microscopy (MFM) [11, 12]. In both methods, EFM and MFM, the cause for the topographic crosstalk is the same namely capacitive coupling effects between tip and substrate due to the mode of operation, the so-called lift mode. EFM and MFM measurements are based on scanning a surface with a tip, oscillating with a frequency near to its resonance frequency in a two-pass technique [13]. In a first scan, the tapping mode, the tip touches the surface in its lowest point thus the topography of the sample becomes visible. In a second scan, EFM and MFM measurements usually are performed in the lift mode at a certain distance from the local sample surface, the so-called lift height, following the topography profile from the first scan. Thus, short-range forces are no longer relevant and the long-range forces such as magnetic and electrostatic forces can be determined. Common to all methods is the

fact that tip and substrate form a capacitor. Due to work function differences of the tip-substrate system, an electrical potential called contact potential difference (V_{CPD}) is present between different materials, leading to electrostatic forces even without an applied tip bias. Thus, changes in distance between substrate and tip due to non-planar structures lead to a decrease in the electrostatic force when the distance increases and an increase in the electrostatic force when the distance decreases, resulting in a significant contribution of topography to the EFM or MFM signal. For MFM measurements, various methods to reduce capacitive coupling contributions are discussed e.g., reducing the tip size or including a dielectric layer between the substrate and the nanostructure as well as changing the tip magnetization [14–16]. In order to investigate the magnetism of nanoparticles, it was shown that while embedding the nanoparticles, the crosstalk due to capacitive coupling disappears [17]. In earlier work [18], an algorithm was developed to correct MFM lift mode data based on a correlation of the AFM signal and the MFM signal. A similar method was developed by van der Hofstadt for lift mode electrostatic force microscopy of non-planar samples [8]. Their algorithm subtracts the contributions of topographic crosstalk from EFM signals. This method was successfully applied to locally determine the dielectric constant of silicon dioxide pillars as well as of single bacterial cells [8]. Many groups determined the dielectric constant of thin films by EFM [1, 5, 19]. Gomila *et al* extracted the topographic crosstalk by subtracting the signal beside the thin film from the signal above the thin film, thus determining the so-called intrinsic capacitance [3]. In EFM exist three possibilities to determine the dielectric constant. The use of the DC-signal and the use of two possible alternating current (AC) signals, the $\Delta\Phi(\omega)$ phase signal and the $\Delta\Phi(2\omega)$ phase signal. In all methods the force is proportional to the first derivative of the capacitance which is related to the dielectric constant. All methods have their advantages and disadvantages. Gramse *et al* reported the advantage of using the DC signal because of no need for additional electronics [1]. Common to all three methods is the topographic crosstalk initiated by the mode of operation, namely the lift mode.

One possibility to avoid topographic crosstalk is the use of the linear mode instead of the lift mode. In this paper we compare linear and lift mode measurements in EFM theoretically, as well as in experiments on various structures. EFM phase signals of dielectric layers with trench-structures reveal that lift mode measurements reduce the sensitivity to measure the dielectric constant significantly, whereas linear mode measurements allow the determination of the dielectric constant of the structured layer. Note that the benefit of linear mode measurements, such as the ability to avoid capacitive coupling, is also resulting in an inflexibility due to the fixed height. Therefore, linear mode can only be used in precisely defined areas. Surfaces with larger lateral structure changes can lead to a lower resolution or even to the destruction of the tip due to surface contact. In principle, the determination of the dielectric constant of nanoparticles is not possible in lift mode measurements, because the change in distance between tip and substrate changes the effective area of the capacitor as proven in [16], so that the contribution of the nanoparticle to the dielectric contrast becomes too small to be detected. In this paper, we determined the dielectric constant of polystyrene (PS), polylactide (PLA) and polymethyl methacrylate (PMMA) nanoparticles with linear mode measurements. It is shown that the contrast in linear mode is independent of the tip form and tip size. In general, it is experimentally proven that the topographic crosstalk often seen in EFM measurements on non-planar nanoscale structures can be avoided by using the linear mode instead of the lift mode, thus allowing the determination of the dielectric constant of nanoscale structures.

2. Methods

Polystyrene (PS), polymethyl methacrylate (PMMA) and polylactide (PLA) particles in a size-range of 80 nm–200 nm were studied regarding the dielectric constant. PS and PMMA nanoparticles were synthesized by miniemulsion polymerization with the non-ionic surfactant Lutensol AT50 to ensure a lower zeta potential [20]. Briefly, 3 g of styrene or methyl methacrylate, 125 mg of hexadecane and 60 mg of the initiator 2,20-azobis(2-methylbutyronitrile) (V59) were mixed together and added to 24 g of water containing 200 mg of non-ionic surfactant Lutensol AT50, which is a poly(ethylene oxide)-hexadecylether with an EO block length of 50 units (BASF). After stirring 1 h for pre-emulsification at 900 rpm, the miniemulsion was prepared by ultrasonicating the mixture for 120 s at 90% amplitude (Branson sonifier W450 Digital, ½″. tip) in ice bath to prevent the polymerization. The polymerization was carried out at 72 °C over night under stirring at 400 rpm.

PLA nanoparticles were prepared by combination of miniemulsion and solvent evaporation methods. Briefly, 0.3 g of PLA were dissolved in 10 g of chloroform and added to 24 g of water containing 72 mg of sodium dodecyl sulfate. After stirring 1 h for pre-emulsification at 900 rpm, the miniemulsion was prepared by ultrasonicating the mixture for 180 s at 70% amplitude in a pulse regime (30 s sonication, 10 s pause) using 1/4″ tip. The obtained miniemulsion was transferred to the 50 ml round bottom reaction flask and left overnight at 40 °C for complete evaporation of chloroform. The obtained nanoparticles were purified by centrifugation to remove the excess of surfactant and characterized in terms of particles size and zeta potential. Zeta potentials were −1 mV, −8 mV and −49 mV for PS, PMMA and PLA nanoparticles, respectively. The nanoparticle

Table 1. Parameters of commercial tips used for measurements: tip radius r, resonance frequency f_r, spring constant k and quality factor Q.

Probe	Company	r/nm	f_r/kHz	k/Nm^{-1}	Q-factor
SSS-MFMR	Nanosensors	15	75	2, 8	190–220
MFMV	Bruker AFM Probes	40	75	2, 8	240–260
MESP-HM-V2	Bruker AFM Probes	80	75	3	240–260

dispersions were diluted with highly purified water (1 μl dispersion with 10 ml water) to avoid aggregation and then dropped 30 μl on a freshly cleaved siegert wafer for drying. Morphological verification of the nanoparticles with regards to sphericity and deformation were performed by TEM and AFM measurements.

All dielectric constant measurements were performed on a Dimension Icon AFM with tips of different radii. The radii ranged from 15 nm up to 80 nm with quadrilateral pyramidic shape, which were validated by scanning electroscope microscopy measurements. Measurements to determine dielectric properties were performed in EFM mode with a scan rate of 1 Hz. Imaging resolution was set to 512 samples per line. The two-pass scan collects topography information using tapping mode and electrostatic information in linear mode with a lift height of 20 nm above the particle. A fundamental limitation of the linear mode is, that the absolute distance between the measuring system and the sample structure cannot be guaranteed.

To calibrate the 20 nm distance between the measuring tip and the spherical sample, the tip was moved closer to the particle in <1 nm steps until direct contact was established. This allowed adjustment to the desired real distance. This step was performed individually for each particle and structure to maintain the constant distance. For calculation and illustration purposes, topography data was extracted as X,Z-data and evaluated by using the data analysis program OriginPro. To verify the acquired data, KPFM measurements were performed to match the VCPD values resulting from the linear mode measurements with those of the conventional KPFM measurements.

The parameters for the calculation of the phase shift are summarized in table 1.

3. Results and discussion

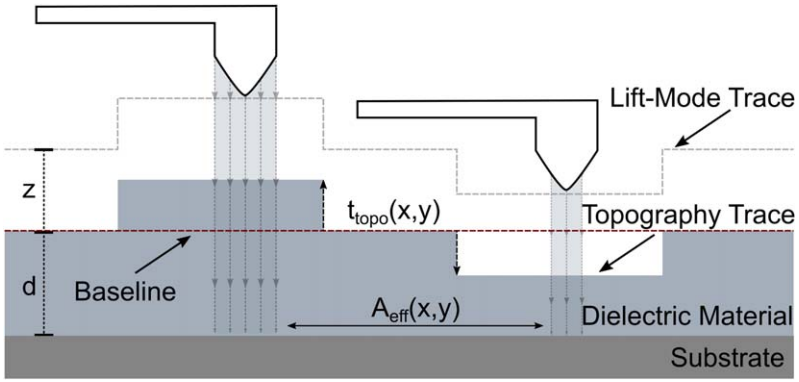
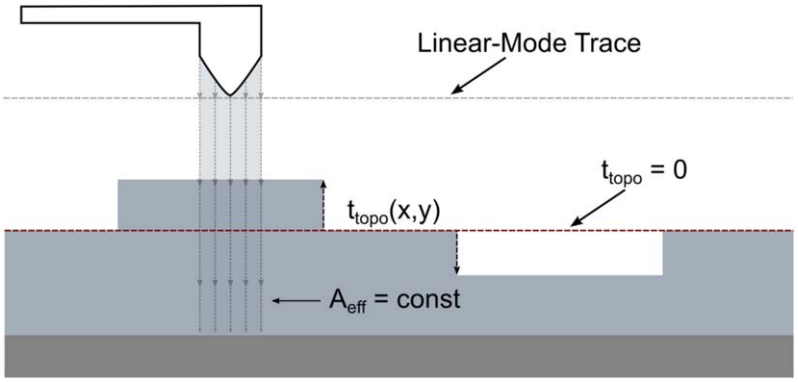
EFM includes different methods to determine the electrical properties, such as the dielectric constant via electrical excitation of the tip by DC or AC voltage. For both, the DC or AC voltage, the force acting on the tip is proportional to the first derivative of the capacitance between tip and sample. Accordingly, all methods are sensitive to changes in capacitance due to changes of the dielectric constant. As mentioned above, EFM measurements can be performed in two modes: the so-called lift mode, following the topography of the first topographic scan in a defined distance as a second scan trace and the so-called linear mode with a fixed distance between tip and substrate. In this paper, we focus on theory and measurements using DC voltage, but the comparison of lift and linear modes is valid for AC signals as well and can be easily transferred to these methods.

3.1. Dielectric contrast of non-planar surface structures in lift- and linear mode measurements

Non-planar dielectric surface structures contribute to the EFM-phase signal in lift mode measurements twice. A first contribution is induced by a change of the volume fraction of the dielectric material in the tip-sample capacitor, forming a dielectric contrast. A second contribution derives from the distance change between tip and substrate following the topography of the sample, as indicated in table 2. This leads to an additional force on the tip, as well as to a change of the effective area A_{eff} of the tip-sample capacitor. Increasing the distance between tip and substrate leads to a positive phase shift corresponding to an repulsive force, whereas decreasing the distance leads to a negative phase shift corresponding to an attractive force [9]. Additionally, A_{eff} has to be adapted as a function of the distance between tip and substrate as described in [9]. As described in our previous works, A_{eff} is defined as the area of an equivalent tip-sample capacitor composed of two circular plates centered on the tip axis [9]. Increasing the distance between tip and substrate increases A_{eff} as reported in [9, 15]. Both contributions are taken into account in the phase shift equation for lift mode measurements in table 2 left column, which gives the phase signal as a function of the topographic parameter and the dielectric constant. In this paper, we use a parabolic tip model as described in our previous works in order to calculate A_{eff} [9, 18]. Measurements in lift mode always include contributions from the topography, reducing the sensitivity to determine the dielectric contrast and even falsifying the results. The influence of the topography decreases for films with non-planar nanostructures with heights small compared to the total thickness of the dielectric layer as discussed in [16].

In contrast to the lift mode, linear mode measurements keep the distance between tip and substrate constant during the measurement, removing the influence of the topography on the phase signal. Therefore, the phase shift resulting from the electrostatic coupling between tip and substrate is only dependent on the amount of

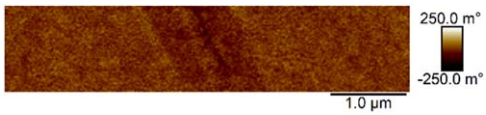
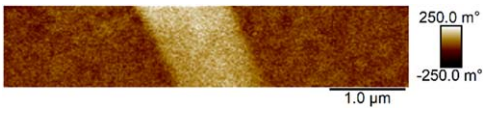
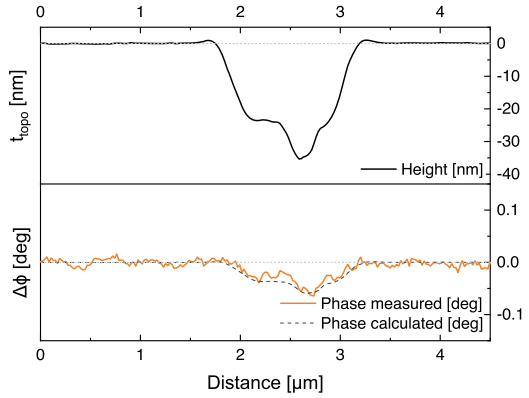
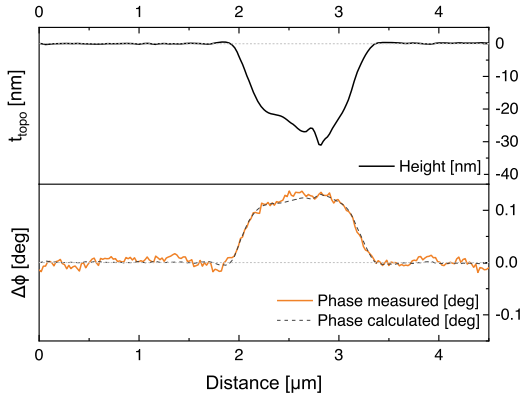
Table 2. Comparison of lift mode and linear mode for non-planar dielectric (nano)-structures and the corresponding equations for the phase shift.

Lift mode	Linear mode
Scheme	
	
Equation	
$\Delta\phi = -\frac{Q}{k}\varepsilon_0 (V_{tot})^2 \left(\frac{A_{eff}(x,y)}{\left(z + \frac{d + t_{topo}(x,y)}{\varepsilon_l}\right)^3} - \frac{A_{eff\,(Baseline)}}{\left(z + \frac{d}{\varepsilon_l}\right)^3} \right)$	$\Delta\phi = -\frac{Q}{k}\varepsilon_0 (V_{tot})^2 \left(\frac{A_{eff}}{\left((z - t_{topo}) + \frac{d + t_{topo}}{\varepsilon_l}\right)^3} - \frac{A_{eff}}{\left(z + \frac{d}{\varepsilon_l}\right)^3} \right)$
$A_{eff}(x,y) = \pi r_{eff}(x,y)^2$ with $r_{eff}(x,y) = \sqrt{\left(\sqrt[3]{\frac{z_1^3}{p}} - z_1\right) r_{tip}}$	
$z_1(x,y) = z + d + t_{topo}(x,y)$	$z_1 = z + d \rightarrow const$

Legend

Q	cantilever quality factor	A_{eff}	effective area of the capacitor	$t_{topo}(x,y)$	topography parameter
k	spring constant	$A_{eff(Baseline)}$	effective area at the baseline	$t_{topo} = 0$	baseline of the topography
ε_0	vacuum dielectric	d	dielectric layer thickness	r_{tip}	tip radius
ε_l	dielectric layer constant	z	lift height	r_{eff}	effective radius based on lift height
V_{tot}	$V_{CPD} + V_{Tip}$	p	percentage factor	(x,y)	position-dependency

Table 3. Comparison of lift mode (left) and linear mode (right) phase shift data for non-planar dielectric (nano)-structures (MFMV tip, lift height 20 nm).

Lift mode	Linear mode
Topography	
	
Phase	
	
Cross-section	
	

dielectric material within the capacitor. Thus, in linear mode measurement the only contrast is a dielectric contrast, whereas in lift mode measurements the contrast has two overlapping contributions, from topography and from the dielectric material in the capacitor reducing the sensitivity for the determination of the dielectric constant. Table 2 shows the differences in the measurement methods and how they affect the calculations of the phase shift and the effective area of the capacitor.

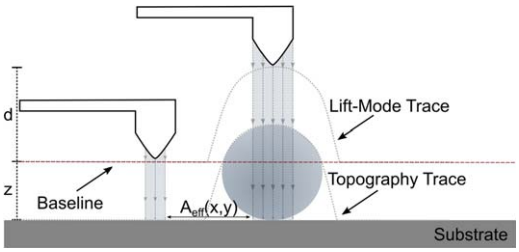
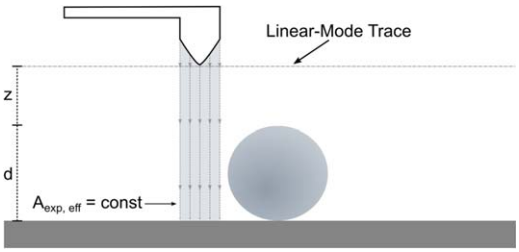
Measurements of thin film structures of 60 nm spin coated ARP-5910 positive-photoresist demonstrate the difference of lift mode and linear mode measurements on non-planar nanostructures. All measurements are performed with a lift height of 20 nm to avoid distortions of short-range forces while still being close enough to detect phase shifts due to material changes.

Table 3 compares lift mode and linear mode measurements of a trench structure with a width of 1.2 μm. In lift mode measurements (table 3 left) we observe a negative phase signal. According to the phase shift equation in table 2 for lift mode measurements (left column), this phase signal consists of a negative contribution due to the decreasing distance between the tip and substrate and a positive contribution due to less dielectric material in the tip-substrate capacitor while measuring above the trench. Considering a trench, the tip is getting closer to its counter plate and, therefore, has a stronger attraction towards the substrate and a respective negative phase. This negative phase signal overlaps the positive phase signal based on the dielectric contrast.

Instead, in linear mode, only the dielectric contrast contributes to the phase signal (table 3 right). Due to less dielectric material in the capacitor the phase signal is positive and larger than the phase signal in lift mode measurements as there is no contrary contribution of the topography. The measured cross-section in the linear mode was fitted by using the respective phase shift equation (right column). We obtained a dielectric constant in the range of 2.9–3.1 for the ARP-5910 resist. This is in accordance with the original value for the dielectric constant provided by Allresist GmbH.

These measurements demonstrate the advantage of using the linear mode to determine dielectric properties of non-planar nanostructures.

Table 4. Comparison of lift mode and linear mode for a single polymer nanoparticle and corresponding equations for the phase shift.

Lift mode	Linear mode
Scheme	
	
Equation	
$\Delta\phi = -\frac{Q}{k}\varepsilon_0 (V_{tot})^2 \left(\frac{A_{eff}(x,y)}{\left(z + \frac{d(x,y)}{\varepsilon_p}\right)^3} - \frac{A_{eff\,(Baseline)}}{(z)^3} \right)$	$\Delta\phi = -\frac{Q}{k}\varepsilon_0 (V_{tot})^2 \left(\frac{A_{\exp}}{\left(z + \frac{d(x,y)}{\varepsilon_p}\right)^3} - \frac{A_{\exp}}{(z+d_p)^3} \right)$
$A_{eff}(x,y) = \pi r_{eff}(x,y)^2$ with $r_{eff}(x,y) = \sqrt{\left(\sqrt[3]{\frac{z_1^3}{p}} - z_1\right) r_{tip}}$	$A_{eff,\exp} = \pi r_{eff,\exp}{}^2$
$z_1(x,y) = z + d(x,y)$	$z_1 = z + d_p \rightarrow const$

Legend

Q	cantilever quality factor	A_{eff}	effective area of the capacitor	p	percentage factor
k	spring constant	r_{tip}	tip radius	$A_{eff,exp}$	experimental half-width area of particle
ε_0	vacuum dielectric	d_p	particle diameter	r_{eff}	effective radius based on lift height
ε_p	dielectric constant of particle	d	topographic particle parameter	$r_{eff,exp}$	experimental half-width radius of particle
V_{tot}	$V_{CPD} + V_{Tip}$	z	lift height	(x,y)	position-dependency

3.2. Dielectric contrast of single nanoparticles in lift- and linear mode measurements

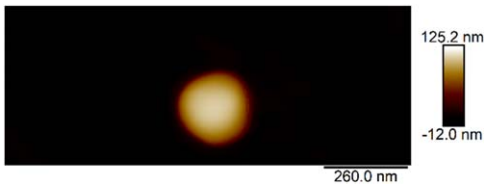
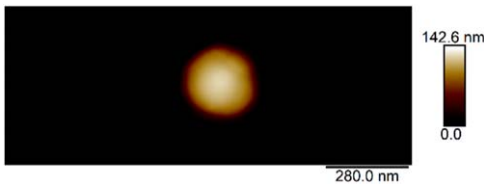
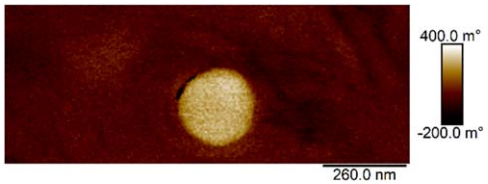
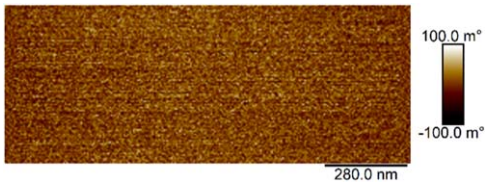
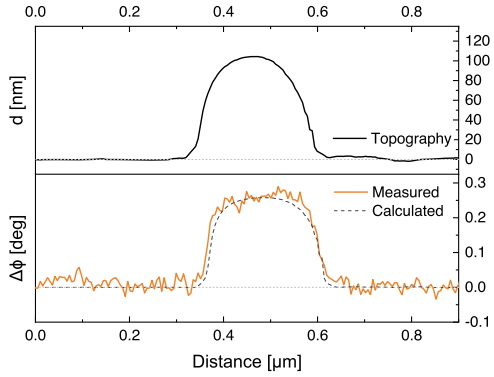
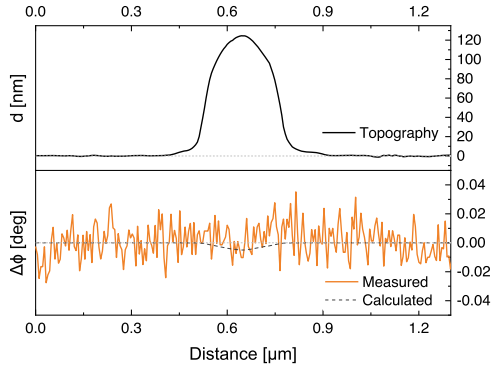
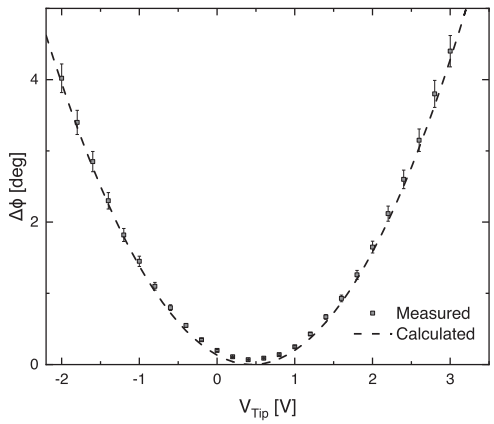
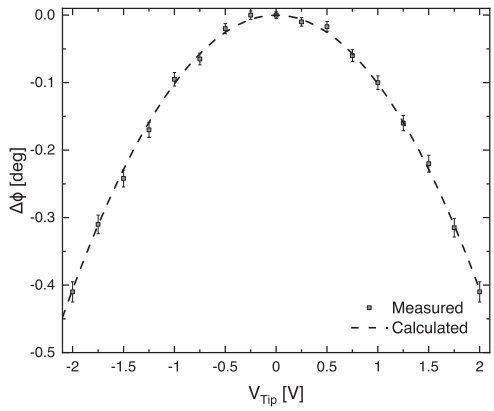
As described above, in lift mode the distance between tip and local sample surface is kept constant during the measurement. Thus, nanoparticles on the surface of the substrate lead to an increasing distance between tip and substrate measuring above the nanoparticles, resulting in a positive phase shift which can be calculated by the respective equation for lift mode measurements in table 4. This distance change induces a topographic crosstalk in the EFM signal. Investigations on the influence of the dielectric constant on the topographic crosstalk showed that the dielectric constant of the nanoparticle has no significant influence because the volume fraction of the nanoparticle in the capacitor is small compared to the volume fraction of the total capacitor [9]. Increasing the particle diameter automatically increases the effective area of the tip-substrate capacitor as well due to the increasing distance between tip and substrate. Thus, the volume fraction of the nanoparticle in the capacitor stays constant and always small compared to the volume of the total capacitor. This effect hinders in principle the determination of the dielectric constant of nanoparticles in lift mode measurements.

Applying an additional external voltage leads to an enhancement of the positive phase signal with a voltage dependence resulting in an upward opened parabola (table 5) caused by the topographic contrast. The contact potential voltage V_{CPD} is given by the vertex of the parabola.

Considering the electrostatic forces for single nanoparticles measured in linear mode, the signal is only due to the contribution of the dielectric constant of the nanoparticle in the tip-sample capacitor, as can be seen in the phase shift equation for linear mode measurements in table 4 (right column). Therefore, the dielectric contrast of the nanoparticle leads to a negative phase shift. Thus, linear mode measurements enable the determination of the dielectric constant of single nanoparticles based on these capacitive effects mentioned above.

In linear mode measurements tip shape and size are less important than in the lift mode as the distance between tip and substrate stays constant. Therefore, $A_{eff,\ exp}$ is a constant pre-factor in the phase shift equation for linear mode measurements in table 4. A_{eff} can be calculated by the parabolic tip model in table 4 left column, used for the calculation of the phase signal in the lift mode [15]. But this equation requires knowledge of the exact value of the tip radius.

Table 5. Comparison of lift-mode and linear-mode phase shift data for a single polymer nanoparticle (MFMV tip, lift height 20 nm).

Lift mode	Linear mode
Topography	
	
Phase	
	
Cross-section	
	
Voltage-profile	
	

In order to determine $A_{eff,\,exp}$ without knowing the exact value of the tip radius the following method of analysis can also be used. Tip size and form are taken into account by using the half-width of the measured topographic cross-section of the nanoparticles, based on the work of Markiewicz et al [21]. The half-width includes the convolution of the tip and the nanoparticle. This method allows to gain $r_{eff,\,exp}$ without knowing the actual tip form and radius. The values obtained are in accordance with the parabolic tip model. The independence of this method from tip size and tip form was demonstrated by comparing three particle groups (PS, PMMA and PLA) with different particle diameter in the range from 80 nm to 200 nm measured with three different tips (see SI).

Table 5 compares lift and linear mode measurements for PS particles. While the phase signal clearly shows the influence of capacitive coupling due to distance changes, linear mode measurements prove that measuring

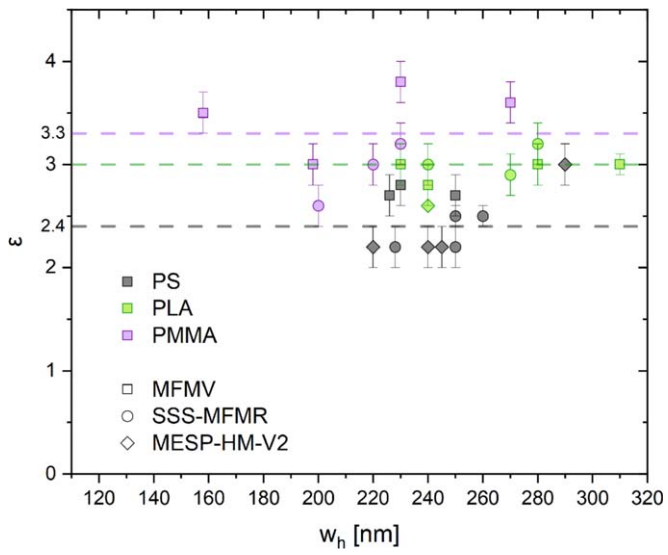


Figure 1. Evaluation of the dielectric constant of nanoparticles of different material and size and of different measuring tips based on voltage profile measurements.

nanoparticles at a lift height of 20 nm above the sample only show a small negative phase shift for V = 0 based on the dielectric contrast. In order to enhance the dielectric contrast, an electrostatic field is applied between tip and substrate in the range of −2 V and +2 V. As this dielectric contrast is directly proportional to the voltage between tip and substrate, an additional external voltage leads to an increase of the negative phase signal resulting in a downwards opened parabola. Analysis of the parabola shown in table 5 measured in linear mode now allows to determine the dielectric constant of single nanoparticles.

3.3. Determination of dielectric constant based on linear mode measurements

In this paper we investigated different polymer nanoparticles made of PS, PMMA or PLA in a size-range of 80 nm–200 nm with three different tips (table 1). The dielectric constant is the only fit parameter in the fit of the parabola at every scanning point. The phase shift calculation is based on the respective equation in table 4 right column. V_{CPD} is determined by the vertex of the parabola, the diameter of the particle by the topographic height and $A_{eff,\,exp}$ determined by the half-width of the topographic cross-section. Figure 1 summarizes the results of these measurements:

The average values of the three materials investigated are $\varepsilon = 2.4 \pm 0.19$ for polystyrene, $\varepsilon = 3 \pm 0.24$ for polylactide and $\varepsilon = 3.3 \pm 0.26$ for polymethyl methacrylate. These values are in accordance with literature values [22, 23].

4. Conclusion

In summary we could demonstrate the influence of topographic crosstalk on lift mode EFM measurements of thin films with non-planar nanostructures. The topographic crosstalk vanishes with increasing dielectric layer thickness between nanostructure and substrate. Topographic contrast can completely be avoided by using the linear mode instead of lift mode for the determination of dielectric properties. It was shown that the dielectric constant of nanoparticles cannot be determined in lift mode measurements. Linear mode measurements allow the determination of the dielectric constant of single nanoparticles independent of tip shape and size. To enhance the contrast, it is necessary to apply a voltage between tip and substrate.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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References

[1] Gramse G, Casuso I, Toset J, Fumagalli L and Gomila G 2009 *Nanotechnology* **20** 395702
[2] Sadewasser S and Barth C 2012 *Characterization of Materials* 1–12
[3] Gomila G, Toset J and Fumagalli L 2008 *J. Appl. Phys.* **104** 024315
[4] Labardi M, Bertolla A, Sollogoub C, Casalini R and Capaccioli S 2020 *Nanotechnology* **31** 335710
[5] Peng S, Zeng Q, Yang X, Hu J, Qiu X and He J 2016 *Sci. Rep.* **6** 38978
[6] Valeriano W W, Andrade R R, Vasco J P, Malachias A, Neves B R A, Guimaräes P S S and Rodrigues W N 2021 *Beilstein J. Nanotechnol.* **12** 139–50
[7] Gomila G, Esteban-Ferrer D and Fumagalli L 2013 *Nanotechnology* **24** 505713
[8] Van der Hofstadt M, Fabregas R, Biagi M C, Fumagalli L and Gomila G 2016 *Nanotechnology* **27** 405706
[9] Fuhrmann M, Krivcov A, Musyanovych A, Thoelen R and Möbius H 2020 *Phys. Status Solidi* (A) **217** 1900828
[10] Tevaarwerk E, Keppel D G, Rugheimer P, Lagally M G and Eriksson M A 2005 *Rev. Sci. Instrum.* **76** 053707
[11] Schwarz A and Wiesendanger R 2008 *Nano Today* **3** 28–39
[12] Jaafar M and Asenjo A 2021 *Applied Sciences 2021* **11** 10507
[13] Vokoun D, Samal S and Stachiv I 2022 *Magnetochemistry* **8** 42
[14] Angeloni L, Passeri D, Reggente M, Mantovani D and Rossi M 2016 *Sci. Rep.* **6** 26293
[15] Krivcov A, Junkers T and Möbius H 2018 *J. Phys. Commun.* **2** 075019
[16] Krivcov A, Ehrler J, Fuhrmann M, Junkers T and Möbius H 2019 *Beilstein J. Nanotechnol.* **10** 1056–64
[17] Krivcov A, Schneider J, Junkers T and Möbius H 2019 *Phys. Status Solidi* (A) **216** 1800753
[18] Fuhrmann M, Musyanovych A, Thoelen R, von Bomhard S and Möbius H 2020 *Nanomaterials* **10** 2486
[19] Arinero R, Riedel C, Schwartz G A, Lévêque G, Alegría A, Tordjeman P, Israeloff N E, Ramonda M and Colmenero J 2009 *Microscopy: Science, Technology, Applications and Education* **3** 1963–77
[20] Musyanovych A, Dausend J, Dass M, Walther P, Mailänder V and Landfester K 2011 *Acta Biomater.* **7** 4160–8
[21] Markiewicz P and Goh M C 1998 *J. Vac. Sci. Technol. B* **13** 1115
[22] Kumar B, Kaushik B K and Negi Y S 2014 *J. Mater. Sci., Mater. Electron.* **25** 1–30
[23] Huber E, Mirzaee M, Bjorgaard J, Hoyack M, Noghanian S and Chang I 2016 *IEEE Int. Conf. on Electro Information Technology* 788–92