SPM 150 Aarhus with KolibriSensor™

Acquisition of atomic site specific force spectroscopy and two-dimensional force maps F(x,z) on KBr(001) and Au(111) at room temperature

Application Notes

- **Site specific force spectroscopy on KBr(001) and Au(111) at room temperature**
- **Acquisition of two-dimesnsional force maps F(x,z) revealing atomic specific contrast**

In non-contact atomic force microscopy (NC-AFM), the forces acting between an oscillating tip and the surface are measured. A topographic image is obtained by keeping the tip sample interaction force (which is measured as the detuning of the resonance frequency) constant. Beyond imaging, force spectroscopy experiments -where the force versus tip sample distance is measured- facilitate quantitative studies of the tip sample forces on the nano-scale. Site specific force spectroscopy is the key to chemical identification of individual surface atoms [1,2,3].

Site specific force spectroscopy experiments are severely hampered by thermal drift especially at room temperature [1,2,3]. The highly symmetrical set-up of the SPM 150 Aarhus allows one to conduct site specific force spectroscopy experiments even at room temperature, due to very low thermal drift rates. Residual drift in X-,Y-, and Z-axis ever present at room temperature was compensated using the Nanonis Atom-Tracking module. Beyond force spectroscopy at specific surface sites, the versatile spectroscopy modules of the Nanonis Control System enable the recording of multidimensional force maps. Furthermore the high stiffness and extraordinary signal-to-noise ratio of the KolibriSensor™ allows imaging and spectroscopy experiments at very small oscillation amplitudes down to A=100 pm for enhanced sensitivity to short-range chemical forces [4].

Force spectroscopy experiments are carried out with the SPM 150 Aarhus with KolibriSensor™ on KBr(001) and Au(111) at room temperature. Measured F(z) curves recorded atop maxima and minima in the topographic images of the KBr(001) and Au(11) surface as well as two-dimensional force maps F(x,z) reveal atomic-specific contrast.

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Force spectroscopy at distinct lattice sites of the KBr(001) surface

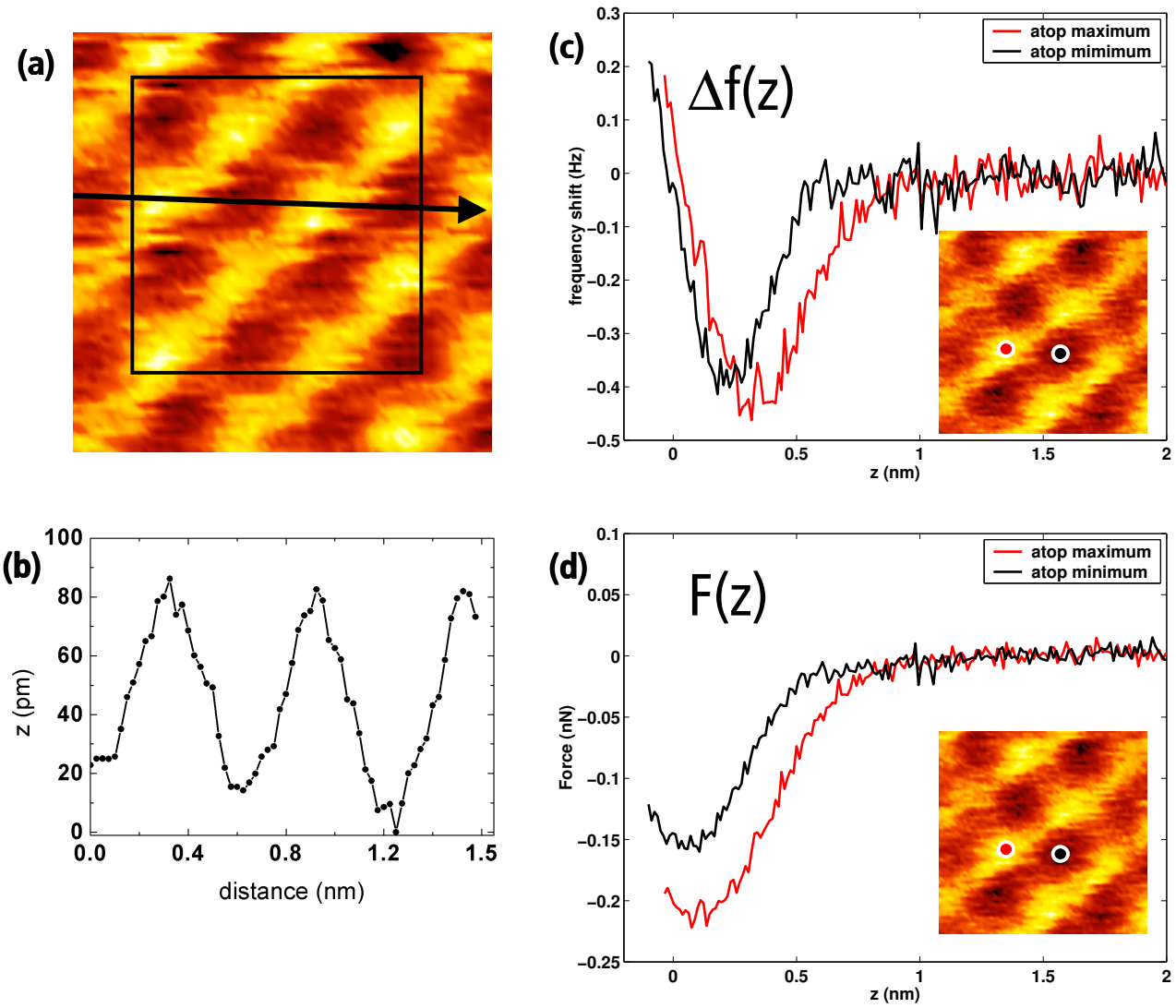

Figure 1: (a) Atomic resolution topographic image of KBr(001) at room temperature. (b) Line profile along the line in (a). (c) detuning versus distance Δf(z) spectroscopic measurements at two positions indicated in the inset image. (d) Calculated [5, see Eq. (1) Appendix] force versus distance curves F(z) for the two Δf(z) curves in shown (c). In comparison a larger attractive force is registered for the position atop a maximum in the topographic image (red curve).

Image size in (a) (1.5 nm x 1.5 nm), f_{res} = 996,033 Hz, A = 100 pm,
Δf = +0.15 Hz, 2 lines/s, 64 x 64 pixels
Image size of inset in (b) and (c) (1 nm x 1 nm), f_{res} = 996,033 Hz, A = 100 pm,
Δf = +0.15 Hz, 2 lines/s, 64 x 64 pixels

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Acquisition of two-dimensional force maps F(x,z) on the KBr(001) surface at room temperature

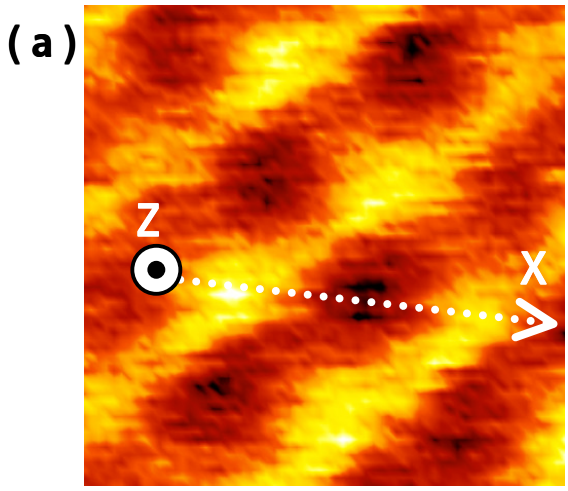
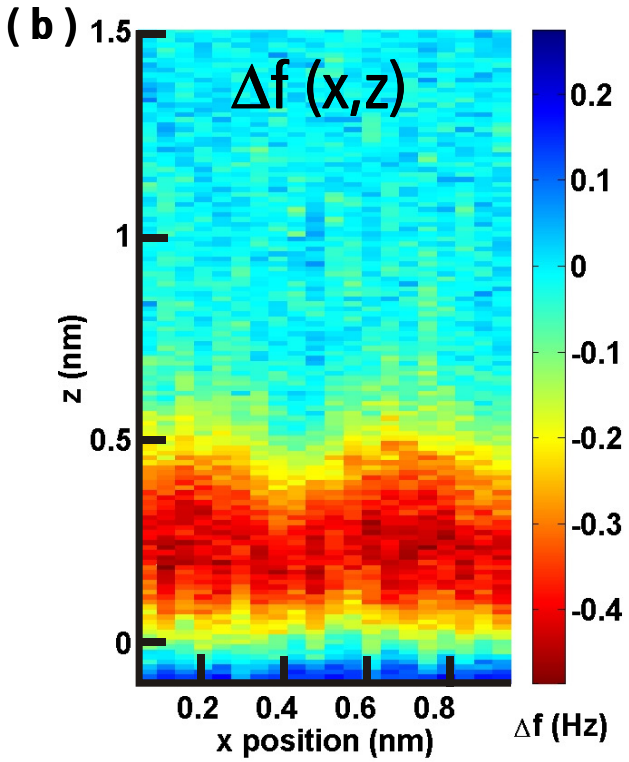
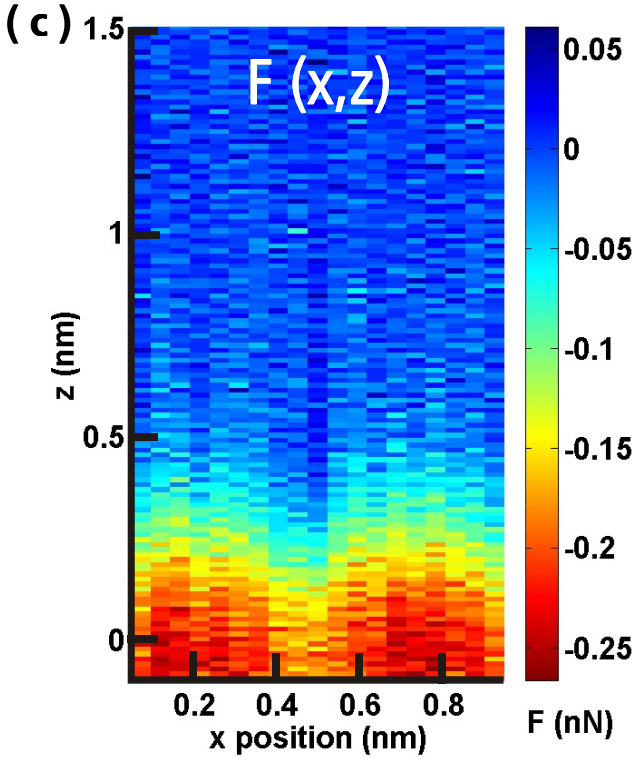


Figure 2: (a) Atomic resolution topographic image of KBr(001) at room temperature. Image size of (a) (1 nm x 1 nm), f_{res} = 996,033 Hz, A = 100 pm, Δf = +0.15 Hz, 2.0 lines/s, 64 x 64 pixels

(b) Measured frequency shift versus relative tip-sample distance (20 curves) as a function of the horizontal tip position Δf(x,z) along the x axis indicated in (a).

(c) The two-dimensional map of the site specific vertical tip-sample forces F(x,z) calculated [5, see Eq. (1) Appendix] from the Δf(x,z) data in (b). A considerably larger attractive force is registered for the positions atop the maxima in the topographic image.





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Acquisition of two-dimensional force maps F(x,z) on the KBr(001) surface at room temperature

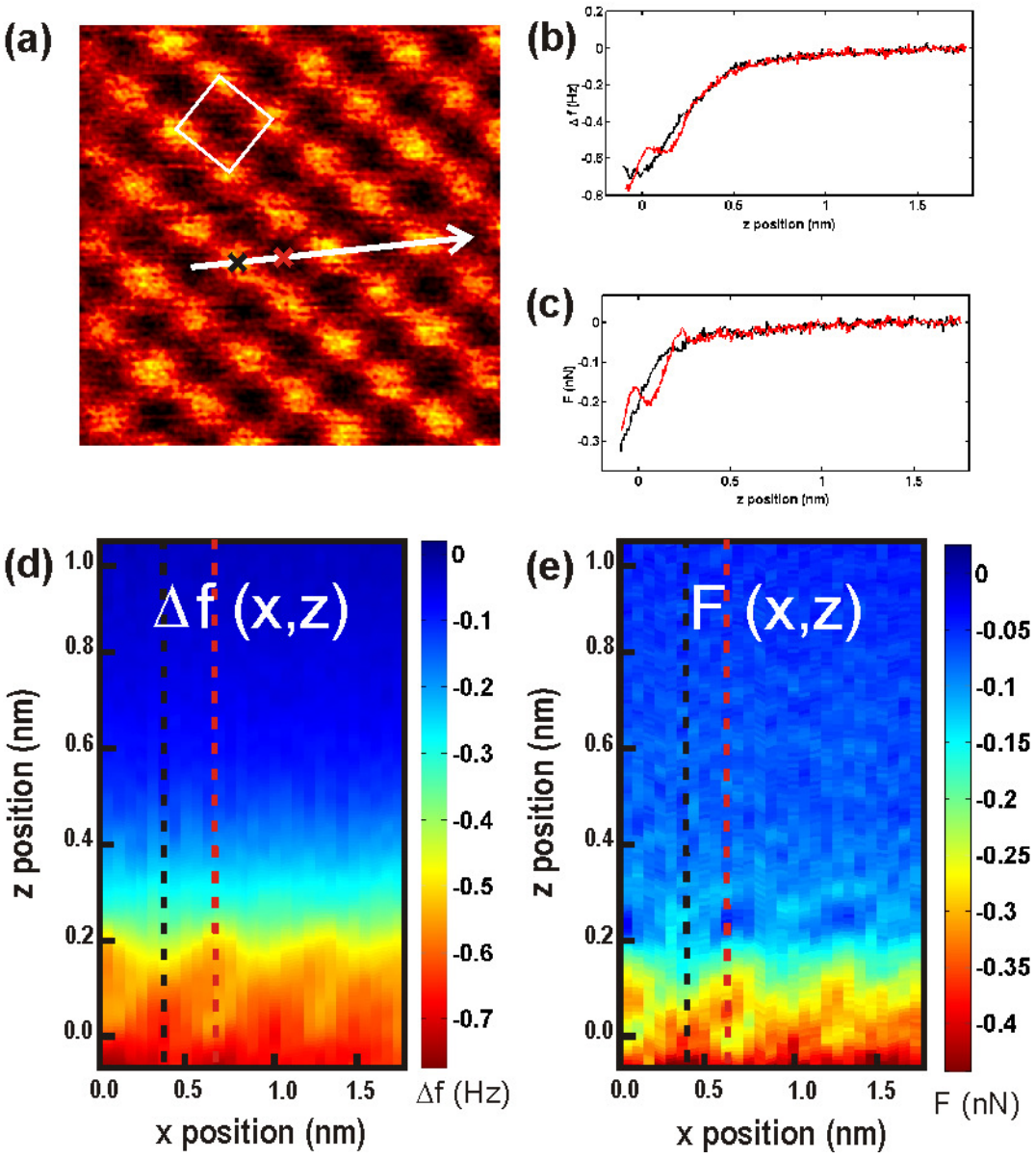

Figure 3: Force map recorded on the KBr(001) surface. (a) Topographic NC-AFM image. (b) Δf(z) and claculated F(z) curves [5, see Eq. (1) Appendix] for the positions indicated by crosses in (a). Curves in (b) and (c) are taken from the recorded (d) Δf(x,z) and derived (e) F(x,z) map at the positions indicated by dotted lines, respectively.

The two-dimensional force map consists of 28 Δf(z) curves featuring 256 data points, respectively. Acquisition time was 1.2 sec/curve. In total 32 sec/force map. (a) imaging parameters: fres= 999, *f*30 Hz, Δfset=-0.37 Hz, A=400 pm,
and Q=28,700. Image size (2,5 x 2,5) nm^2, 256 x 256 pixels, raw data.

Acquisition of two-dimensional force maps F(x,z) on the Au(111) surface at room temperature

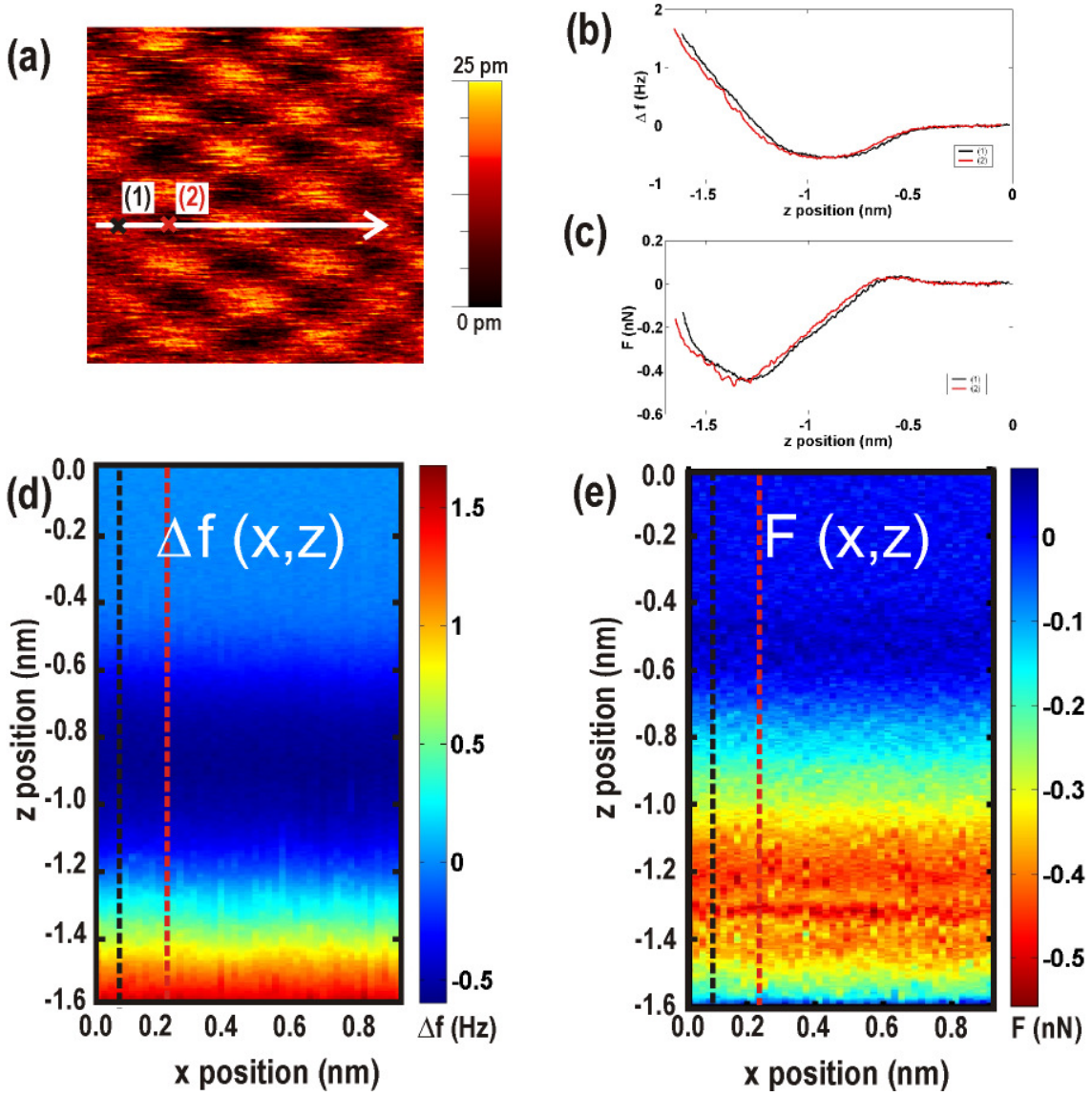

Figure 4: Topographic NC-AFM image of the Au(111) surface. Two Δf(z) curves and derived [5, see Eq. (1) Appendix] force distance curves F(z) recorded atop a maximum (1) and minimum (2) position in (a) are presented in (b) and (c), respectively. (d) and (e) show Δf(x,z) and derived F(x,z) maps .

No tunneling current was detected throughout the experiment.

Imaging parameters: (a): A = 400 pm Df_{set} = +1.24 Hz, U_{CPD} = + 0.6 V, 1.1 nm x 1.1 nm, (256 x 256) pixels, imaging speed: 6.6 lines/s, raw data.

Spectroscopy parameters: 1.6 nm z-sweep distance, 46 curves, 256 points/curve,1.3 s/curve

Acquisition of two-dimensional force maps F(x,z) on the KBr(001) and Au(111) surface

All displayed data have been recorded with no external dampers for the UHV system at SPECS' Laboratory.

Displayed images represent raw data with no filtering or smoothing applied.

Measurement: S. Torbrügge, SPECS GmbH

References

[1] Chemical identification of individual surface atoms by atomic force microscopy, Sugimoto, Y., et al., Nature (London) **446**, 64 (2007)

[2] Sublattice Identification in Scanning Force Microscopy on Alkali Halide Surfaces Hoffmann, R., et al., Phys. Rev. Lett., **92**, 146103 (2004)

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[5] Accurate formulas for the interaction force and energy in frequency modulation force spectroscopy Sader, J. E. and Jarvis, S. P., Appl. Phys. Lett., **84** (10), 1801 (2004)

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Appendix:
Calculation of the tip-sample interaction force F(z) from frequency shift data Δf(z)

In order to derive quantitatively the interaction force F(z) acting between tip and sample from the measured frequency shift Δf(z) data, a non-trivial numerical inversion is required.

This inversion procedure depends on the actual oscillation amplitude of the force sensor during the spectroscopy experiment. Recently, in literature an accurate formula for the calculation of the interaction force from frequency shift data was introduced [5]. According to *Sader and Jarvis* the tip sample interaction force F(z) can be calculated for **any oscillation amplitude** by the expression:

$$F(z) = 2k\int_z^\infty \left(1+\frac{\sqrt{A}}{8\sqrt{\pi(t-z)}}\right)\Omega(t) - \frac{A^{3/2}}{\sqrt{2(t-z)}}\frac{d\Omega(t)}{dt}dt \qquad (1)$$

with $\Omega(z) = \Delta f(z) / f_{res}$,the oscillation amplitude A (A=100 pm for the experiments shown here), and spring constant k (k=540000 N/m for the KolibriSensor™).

In the case of the **small oscillation** approximation it is assumed that the oscillation amplitude is so small that the measured detuning value remains constant over the whole oscillation cycle [5]. Thus it is straightforward to calculate the force acting between the tip and sample in this case by the expression

$$F(z) = 2k\int_z^\infty \Omega(t)dt \qquad (2)$$

Thus, working at small oscillation amplitudes results not only in a higher sensitivity for the detection of the short range chemical interaction between tip and sample but also simplifies the quantitative analysis of force spectroscopy data. *However, this formula is only valid if the assumption of a constant Δf signal over the entire oscillation cycle is fulfilled.*

In the following the quantitative calculation of F(z) according to both formulas is compared.

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Appendix:
Calculation of the tip-sample interaction force F (z) from frequency shift data Δf (z)

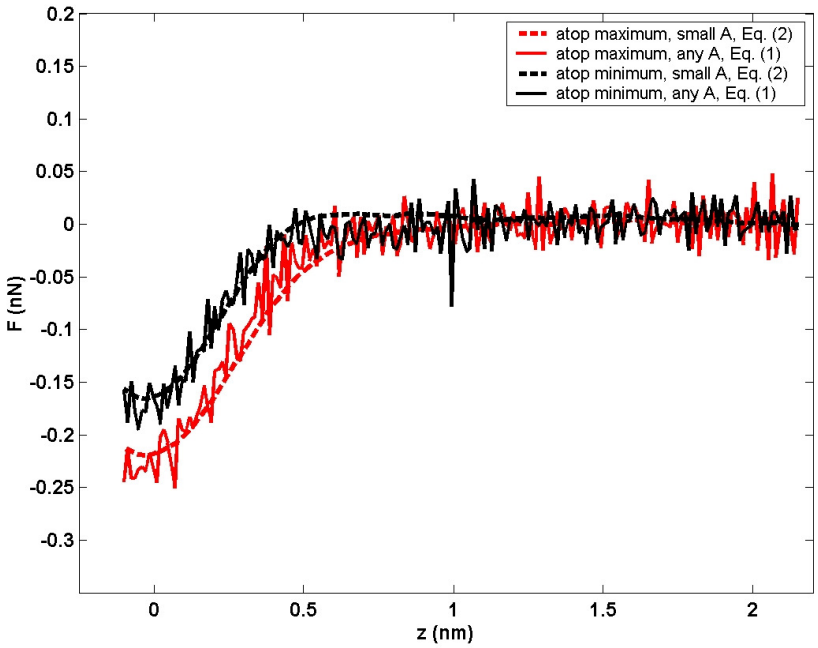

Figure 3: Comparison of the calculated forces F(z) using equations (1) and (2). The forces have been calculated from the frequency shift curves presented in Figure 1(b).

In this case the forces calculated with the small amplitude approximation formula coincide with the forces using the formula which is valid for any oscillation amplitude. From this it can be concluded that the small amplitude approximation is valid for the quantification of the experimental data obtained using an experimental oscillation amplitude of A=100 pm.