Near-zero contact force atomic force microscopy investigations using active electromagnetic cantilevers

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Abstract
Atomic force microscopy (AFM) belongs to the high resolution and high sensitivity surface imaging technologies. In this method force interactions between the tip and the surface are observed to characterize sample properties. In the so-called contact AFM (C AFM) mode the tip is brought into continuous contact with the sample. Significant progress in the AFM technology can be obtained, when the so-called active cantilever technology is implemented in the surface measurements. The built-in deflection actuator enables very precise excitation of the cantilever. Moreover, as the mass of the beam is very small the static beam displacement can be controlled in the wide frequency range. In the experiments, which we describe in this article, we applied the so called active electromagnetic cantilevers. They integrate a conductive loop which, when immersed in the magnetic field and biased with electric current, acts as an electromagnetic deflection actuator. The induced and precisely estimated Lorentz force, which is a function of bias current, cantilever geometry and magnetic field makes the cantilever deflect. Moreover, the probe stiffness can be calibrated with lower uncertainty as in the case of standard thermomechanical analysis. NZ AFM technology required application of a novel control algorithm, called PredPID, in which the cantilever bending caused by a proportional-integral-derivative (PID) block maintaining the constant load force was predicted.

Keywords: AFM, active cantilever, zero force, adhesion force, electromagnetic cantilever

(Some figures may appear in colour only in the online journal)

1. Introduction
Atomic force microscopy (AFM) is one of the high resolution and high sensitivity surface imaging technologies. In this method, force interactions between the tip and the surface are measured to characterize sample properties [1]. In general, two basic AFM technologies can be distinguished. In the so-called contact AFM (C AFM) mode the tip is brought into continuous contact with the sample. The repulsive interactions stemming from the overlap of the tip and surface atom orbitals cause a static cantilever deflection. In intermittent mode (IM AFM) the tip senses the force interactions at the cantilever resonant frequency. Reliable surface imaging requires precise control of the interactions acting at the tip. Only in this case can tip and/or sample modifications be avoided and quantitative (in other words metrological) description of the sample properties be made possible. The advantage of the IM AFM based technologies are relatively simple implementation and surface imaging using cantilevers with a resonant frequency of several kilohertz, which permits a relatively high surface scanning speed. Unfortunately, quantitative analysis of the force interactions between the probe and the investigated sample seems very difficult or even impossible [2]. In contrast, the interactions in Contact-Mode AFM (CM AFM) can be described with far greater accuracy, which is basically related to the linear formula describing the load force. This is related to the nonlinear...
effects occurring when the tip touches the surface for only part of the resonance cycle. Moreover, the cantilever movement is a function of unknown viscoelastic surface properties that are difficult to describe and interpreted.

At the same time, in C AFM the determination of the load force \( F \) acting at the tip is very reliable and is based on the equation \( F = k z \), where \( k \) is the cantilever stiffness and \( z \) is the cantilever deflection. Procedures for determination of the stiffness have been proposed by many groups [3]. From the point of view of measurement of the technological samples, calibration methods of higher throughput than e.g. focused ion beam (FIB)-based technologies are needed. In this context virtual technologies are of great interest, as they enable comparison of the results obtained using various technologies [4]. Many technologies have also been proposed for calibrating the output of the cantilever deflection detector. In the case of optical beam deflection (OBD) detectors, the techniques proposed by Labuda et al are among the most commonly-used for the determination of the so-called inverted optical beam lever sensitivity (InvOLS) [5]. However, the drawback of these technologies is that they require a cantilever test deflection which is usually done by pushing the cantilever against a hard substrate. This can lead to tip damage and can reduce the resolution of the subsequent surface imaging.

It can also be noticed that surface measurements in which the load force is reduced below one nN are becoming more and more attractive [6]. A low load force regime is important for the investigation of biological samples and two-dimensional (2D) materials but in this case, it is really important not only to exert but also to control and measure the load force. Until now, low force experiments have usually been carried out by silicon nitride or polymer probes, as these cantilevers exhibit small stiffness because of their low thickness. Unfortunately, because of microfabrication features, e.g. difficulties in the control of the structure geometry and unknown mechanical parameters, the repeatability of the sensor properties is limited and the calibration of the sensor characteristics should be done for every sensor separately. In this case surface imaging can be executed with a high resolution even if the load force control is performed with limited accuracy. As an alternative to soft cantilevers dedicated to C AFM, one can mention some hybrid modes such as PeakForce Tapping by Bruker [7]. In this mode, the IM AFM probe intermittently contacts the sample at a frequency well below the cantilever resonant frequency, which results in improved contact force control. Despite the low force measurement capability, the tip-sample force and distance vary periodically, which makes the contact force hard to estimate explicitly.

So-called piezoresistive technology offers a very reliable solution, in which the cantilever incorporates a piezoresistive deflection detector [8]. The change of its resistance corresponds with the cantilever deflection and therefore the response of the beam can be calibrated as a function of the force acting at the tip or the beam deflection, independently of the system in which the experiments are to be performed [9]. The stiffness of the piezoresistive cantilevers is relatively high, which makes them an excellent solution for resonance applications. Furthermore, C AFM experiments are feasible, but in order to avoid tip wear and surface modification, the application of advanced and precise control algorithms is required [10].

Significant progress in AFM technology can be made when so-called active cantilever technology is implemented for surface measurements. Active cantilevers integrate probe displacement actuators. Built-in deflection actuators, in contrast to the setup in which external shakers are used, enable very precise excitation of the cantilever resonant vibration only. Moreover, as the mass of the beam is very small, static beam displacement can be controlled in a wide frequency range (up to the structural resonance frequency which is usually tens of kHz).

Structures with piezoelectric actuators belong to the group of active cantilevers [11]. An additional piezoelectric layer deposited on the spring beam increases the structure stiffness, which reduces the range of the static probe deflection and as a consequence, C AFM applications. Moreover, the excitation of the resonant vibration requires the actuator to be biased with a relatively high voltage, which is impractical in many applications.

The tip movement can also be induced electrothermally, when the temperature in the microheater is modulated. In this case, due to the difference in the thermal expansion coefficients of the materials forming the structure, resonance vibrations of up to 300 kHz frequency were presented at a dissipated power of up to five mW [12].

In the experiments which we describe in this article, we applied so-called active electromagnetic cantilevers. These integrate a conductive loop which, when subjected to a magnetic field and biased with an electric current, act as an electromagnetic deflection actuator. The induced and precisely estimated Lorentz force, which is a function of the bias current, the cantilever geometry and the magnetic field makes the cantilever deflect. In this way, as all these factors can be precisely determined, the probe stiffness can be calibrated with lower uncertainty, as in the case of standard thermomechanical analysis [13]. It should be noted that our calibration procedure is based on applying the electromagnetic test force at the cantilever apex. The test force can be explicitly calculated and easily modulated over a wide range, making it possible to test not only the detector sensitivity but its nonlinearity as well. Furthermore, based on the electromagnetically calibrated cantilever stiffness, it is possible to determine the InvOLS without having to press the probe against a hard reference substrate.

Here we also describe experiments in C AFM investigations, in which the load force at the tip was kept constant by control of the current biasing the Lorentz loop of the electromagnetic cantilever. As the control was done with high precision and the beam stiffness was low, it was possible to reduce the load force even to the negative range; the proposed technology is called the near zero AFM (NZ AFM) mode. In this case the tip-sample contact was given by the interaction determined by the snap-in force, which was observed when the tip approached the surface under investigation. When microbiological specimens are imaged, these interactions can be influenced by a liquid film covering the measurement object. Therefore, the precise control of the load force between the tip and the sample is really crucial, so as not to modify the imaging
conditions. The same applies for measurements of suspended membranes of two dimensional (2D) materials, where a large load force acting at the tip can lead to damage to the measured object.

NZ AFM technology required application of a novel control algorithm, called PredPID, which predicted the cantilever bending caused by a proportional-integral-derivative (PID) block maintaining a constant load force.

In the PredPID scheme, as InvOLS was determined, it was possible to record the topography by analysing the output of the OBD detector. The InvOLS was calibrated by test cantilever deflection in the range of ±1.6 µm, the stiffness of the electromagnetic cantilevers was in the range of tens of mN m⁻¹ and the resonant frequency was several kHz. The electromagnetic cantilever was subjected to a magnetic field of 330 mT and the bias current was in the range of ±2 mA. For the surface investigations, FIB-milled diamond tips with a diameter of 50 nm and a height of five µm were used. The diamond tips were mounted on the cantilever using focused ion beam induced deposition (FIBID) technology which ensured the needed precision and durability.

The developed setup made it possible to observe the sample topography of a silicon reference structure at a load force of 100 pN and a scanning frequency of twoHz in NZ AFM mode. The topography of a highly oriented pyrolytic graphite (HOPG) surface was observed, where the atomic edges were clearly identified, proving that the proposed NZ AFM technology exhibited the expected resolution.

2. NZ AFM mode

2.1. Electromagnetic actuation

Active electromagnetic cantilevers, which were U-shaped conductive cantilevers fabricated using the technology presented in [14], were subjected to a magnetic field and driven by a Howland current source (HCS) (figure 1). The general formula for the electromagnetic force induced by the bias current in the magnetic field is given by:

$$\vec{F} = (\vec{i} \times \vec{B})L$$

(1)

where $\vec{i}$ is the bias current, $\vec{B}$ is the inductance of the magnetic field and $L$ is the effective length of the conductive loop segment perpendicular to the magnetic field lines. When the field is uniform and directed along the $X$-axis, the above formula can be rewritten as:

$$F_{Lz}(t) = i(t)B_{x}L,$$

(2)

where $F_{Lz}$ is the electromagnetic force induced in the cantilever $Z$-axis, $B_{x}$ is the magnetic induction in the $X$-axis and $i(t)$ is the instantaneous current biasing the cantilever. The formulas above indicate that the resonance and the static cantilever deflection can be controlled with high precision by modulating the cantilever bias current.

In such a solution, the interaction force acting at the cantilever tip is controlled by the Lorentz force and not by the $Z$-axis scanner applied in the standard AFM experiments. Thanks to the cantilever’s very low stiffness ($k = 0.027 \text{ N m}^{-1}$), it was possible to achieve a high range of deflection, up to ±1.6 µm. Due to the fact that the cantilevers were, at the same time, very long (500 µm), it was possible to assume that for small deflections (e.g. <five µm) the tip at the very end of the cantilever was only moving along the $Z$-axis.

2.2. PredPID control loop

In the standard AFM system, the force interaction causes cantilever vertical deflection (VD) which is observed by a dedicated displacement sensor. In order to maintain a constant interaction at the cantilever tip, a feedback loop is applied. The VD signal is subtracted from the so-called set point (SP) to form the control error (CE) signal. The controller, usually operating as a PID block, sets its output control variable (CV) to the null CE signal. This is conducted as the CV signal modulates the length of an external piezoeactuator defining the tip vs. surface position.

In the active cantilever setup, the VD signal contains entangled information about deflections caused by the on-cantilever deflection actuator and induced by the tip-sample interactions. In order to control the interactions at the tip, the response of the cantilever deflection detector as a function of the actuator operation must be predicted. In PredPID, the complete CV signal is transformed in the actuation force prediction block to calculate the signal component corresponding with the actuator operation as shown in figure 2(b). Based on the known InvOLS it is possible to estimate the cantilever actuation deflection for any given CV value. In the force compensation block, the component corresponding to the actuation is subtracted from the complete VD signal, scaled by the cantilever stiffness. As the result the component corresponding to the contact force (CF) can be determined, which is then subtracted from the force set point (FSP) to calculate the force.
control error (FCE). The standard PID controller is used to null the FCE by setting the CV signal.

The proposed PredPID control technique is a deterministic one, because the entire system can be modelled in a reliable and repeatable way based on the well-described deterministic characteristics. This involves an InvOLS calibration, which in our case is done without pressing the cantilever against the investigated surface [15]. Moreover, the operation of the force compensation and actuation prediction blocks is of great precision, as the electromagnetic actuation is linear vs. the bias current which is indicated by equations (1) and (2) (figure 2).

2.3. Halbach array

In order to obtain linear movement of the electromagnetic cantilever and determine the InvOLS, the magnetic field needed to excite the Lorentz force should be uniform, well-known and directionally-adjustable. In our setup the sample and the electromagnetic cantilever were placed in the centre of an array of permanent NdFeB magnets of N45 grade, which formed a so-called Halbach configuration [16] shown in figure 3(a). A magnetic induction of 330 mT was measured inside the Halbach array along the uniformity axis around the cantilever and less than 3 mT was measured in the other axes, with an uncertainty of ±1 mT. The angular position of the Halbach array can be adjusted in the range of up 30° as shown in figure 3(b).

2.4. Near zero atomic force microscope

In the investigations performed, a home-made atomic force microscope was used, operating in ambient conditions equipped with a so-called ARMScope controller [17]. Figure 4 shows the measurement head. The applied machine was a bottom, open loop scanner system which used an OBD displacement detector to monitor the cantilever displacement. Its modularity made it possible to adapt the system architecture to the NZ AFM mode. The scanner’s range of movement was $20 \times 20 \times 5 \mu m^3$ in the X, Y and Z directions, respectively. As in the experiments performed where active electromagnetic cantilevers were used, a Z-axis scanner was not applied to control the distance between the probe and the sample. All the scans were recorded with a resolution of 512 by 512 pixels using a piezoelectric X-Y scanner over a $5.5 \times 5.5 \mu m^2$ area with a scan speed of up to two lines s$^{-1}$.

The developed PredPID controller was implemented using mixed analogue-digital electronics. The predictive loop and force compensation modules were analogue circuits and the entire PID algorithm was implemented in an ARM-type microcontroller, which was coupled with an 18-bit analogue to digital converter (ADC) achieving 109 dB SNR and a 16-bit digital to analogue converter (DAC) with 96 dB SNR and a bandwidth limited to 38 kHz. Such a platform guaranteed accurate PredPID scheme implementation in the NZ AFM investigations.

3. Active electromagnetic cantilever design

3.1. Cantilever fabrication

An active cantilever optimized for electromagnetic actuation [18, 19] was fabricated on the basis of a double-sided micromachining concept [20]. A silicon-on-insulator (SOI) n-type wafer with a 400 µm thick handle layer, a one µm thick buried oxide and a 1.5 µm thick device layer was used as the input substrate (figure 5(a)). At the beginning, a 100 nm thick silicon dioxide layer was thermally grown in a wet oxidation process—(figure 5(b)). As the next step, the silicon dioxide layer was removed from the front side. In the third step, using a low-pressure chemical vapour deposition (LPCVD) method, the device layer was p-doped (figure 5(c)). The applied LPCVD process [21] allows a homogeneous, high boron concentration of $1 \times 10^{20} \text{cm}^{-3}$ to be obtained. Next, after the deposition of the gold layer, mirrors and contact paths were defined by a photolithography process (figure 5(d)). In the post-CMOS sequences, the shapes of the cantilevers were defined by a front side plasma etching followed by a photolithography process (figure 5(e)). It is worth emphasizing that the use of the SOI-type substrate guaranteed that cantilevers with a precisely defined thickness were obtained (the final cantilever thickness depends exclusively on the thickness of the SOI device layer). Next, after sputtering a 100 nm thick aluminium layer on the back side of the wafer, followed by back-side photolithography and wet etching of the aluminium, the overall shape of the support...
The NZ AFM setup—measurement head.

3.2. Tip preparation

The active electromagnetic cantilevers used in our experiments were fabricated as tipless structures. In order to conduct NZ AFM experiments, diamond tips were mounted at the beam apexes. Diamond particles sized ca. $15 \mu m$ were dispersed in ethanol and placed on a silicon transfer substrate with a pipette. After evaporation of the alcohol (in air conditions), the active electromagnetic cantilevers and the prepared transfer substrate with the particles were transferred to a scanning electron microscope and focused ion beam (FIB) system—FEI Helios NanoLab 600i. Slender type particles were selected as particles for forming the future tip. The so-called EasyLift micromanipulator was placed close to the identified particle and attached using a focused electron beam induced deposition (FEBID) process.

Rotation of the EasyLift micromanipulator made it possible to place the diamond perpendicularly to the cantilever surface as shown in figure 6(a). The tip was fixed to the cantilever by a film deposited by the focused ion beam induced deposition (FIBID) process. In both the FEBID and FIBID processes a trimethylcyclopentadienyl precursor was applied and the films fixing the diamond particle to the manipulator and cantilever were formed by a carbon matrix with platin nanograins [22].

As the film deposition rate in the FIBID process is higher than the deposition rate in the FEBID process, it was possible to detach the EasyLift manipulator from the diamond particle by breaking the first bond. The tip apex of the diamond particle was quite large and irregular, therefore the FIB process was used to form the final probe shape (figure 6(b)) in two steps:

1. Coarse milling—to form an outline on the tip, an ion beam current of around ten nA and a short dwell time (below ten us) should be used,
2. Fine milling—to ensure the best wall quality and a small tip radius, a small ion current should be used (around 100 pA); a dwell time longer than 100 us provides better wall quality.

Application of bulky diamond nanotips is very advantageous in NZ AFM experiments as the diamond ensures the highest possible durability against wear, and the height of ca. ten $\mu m$ makes it possible to access the deeper structures on the investigated surface.
Figure 5. Fabrication of active electromagnetic cantilevers on the device layer of a SOI substrate.

Figure 6. (a) Placing the diamond particle on an active electromagnetic cantilever using EasyLift manipulators. (b) The tip after the shaping process.

4. Results

4.1. Active electromagnetic cantilever characterization

The properties of the active electromagnetic cantilevers were determined using a commercial SIOS NVA (Nano Vibration Analyzer) precision laser vibrometer, which was combined with an arbitrary signal function generator (Tektronix AFG3021), an HCS running at up to two MHz with a maximum output current of 10 mA and a data acquisition system. The system described here made it possible to analyse a cantilever displacement in the frequency range of up to one MHz with a resolution of 100 fm/√Hz. The measurement setup is shown in figure 7.

4.2. Measurements of the static and resonant active electromagnetic cantilever deflection

In the first characterization step of an active electromagnetic cantilever, its thermomechanical vibration noise was recorded using a laser vibrometer (8). In this way, the cantilever stiffness, resonant frequency and quality factor were calculated. Moreover, it was possible to determine the InvOLS but only with an uncertainty of 15% [3, 23]. The spring constant of the cantilever calculated from the thermal noise curve was equal to $0.027 \pm 0.004$ N m$^{-1}$.

In the second characterization step, the cantilever was deflected by a Lorentz force, which made it possible to improve the characterization uncertainty. In this step, the
Figure 7. The vibrometer setup for investigating cantilever mechanical properties and also linearity of deflection.

Figure 8. The thermomechanical noise measurement of an active electromagnetic cantilever obtained with a vibrometer (black points) and an applied OBD measurement head (red points). The continuous lines represent the respective resonance curve fits.

cantilever displacement was measured by the laser vibrometer and the beam stiffness was determined as the ratio of the cantilever apex deflection and the applied electromagnetic force. As the bias current, Lorentz loop and magnetic field were described with high precision, the uncertainty of the stiffness calibration in this method was five% [13]. In this case, the stiffness of the applied cantilever was determined to be 0.037 ± 0.001 N m\(^{-1}\). The stiffness obtained in this way was higher than the stiffness calculated on the basis of the analysis of the thermomechanical noise. The difference resulted from the fact that the electromagnetic force acted as a point-type load, whereas in the thermomechanical analysis, the cantilever was loaded uniformly by particle fluctuations.

The minimal force which can be actuated was limited by the thermomechanical vibration noise of the active cantilever and was 30.15 fN \(\sqrt{\text{Hz}}\), when calculated as described in [21].

When a Lorentz loop is biased, parasitic thermomechanical actuation can also occur [19]. In general, as the thermomechanical actuation depends on the bias current squared (in other words, on the power dissipated in the cantilever) its influence becomes significant at a higher bias current. In NZ AFM experiments the active cantilever should be actuated electromagnetically, thus the range in which the probe should be displaced linearly must be precisely estimated as a function of the driving bias current. In this case, the total harmonic distortion (THD) of the output signal of the interferometer was analysed when the cantilever was actuated in the magnetic field [12]. In the next step, the experiment described above was repeated and the THD of the OBD detector output signal was analysed.

This allows us to not only assess the linearity of the OBD setup, but also to calibrate its InvOLS [15], which in our case is 245 nm \(V^{-1}\). A comparison of the recorded THD curves shows that the THD of the OBD detector is higher than the THD analysed with the laser vibrometer. This is related to the fact that the OBD setup was sensitive to the angular structural deflection but not the vertical beam motion. Moreover, the four-quadrant OBD head response always became always nonlinear when the laser beam reflected from the cantilever illuminated the upper or bottom sections of the photodetector, which was the case when the cantilever was heavily displaced.

4.3. NZ AFM surface measurements

In order to present the capabilities of the proposed NZ AFM technology, several surface imaging investigations were performed. In the first experiments the topography of a TGQ1 reference sample was measured. This had a SiO\(_2\) 20 nm thick square grating with planar dimensions equal to 1.5 \(\mu\)m \(\times\) 1.5 \(\mu\)m with a 3.0 \(\mu\)m period on a silicon substrate [24]. In this way it was possible to calibrate an AFM X-Y scanner, as well as to determine the PredPID control parameters, define the tip scanning speed and estimate the range of the load force acting at the tip. During the experiment, the following signals were recorded (names according to figure 2(b)):

- the VD signal, which is equivalent to the surface topography as detected by a standard atomic force microscope,
- the CV signal, which controls the Lorentz force (equation (2)) needed to maintain a contact force equal to the force set point,
- the CF signal, which is equal to the actual tip-sample interaction force.

The value of the load force was estimated based on the recorded data from the active electromagnetic cantilever, with a force-distance (F-z) curve indicating the repulsive, adhesion and snap-in forces—see the curves shown in figure 9.

In the first scanning experiment the reference sample was imaged with a set point load force of 2.80 nN, which corresponds with standard contact AFM imaging. During the surface scanning the contact force varied from 2.68 nN up to 2.96 nN (figure 10). The exact quantitative determination of the contact force was possible because of the known characteristics of the
active electromagnetic cantilever and the known InvOLS of the OBD detector.

In the second scanning experiment, a scan with a set point contact force equal to zero nN was performed. In this case, the distance between the tip and the sample was maintained so that the attractive and repulsive forces were equal. Any deviation from the defined set point was compensated for by the Lorentz force set by the PredPID controller. In figure 11(c) the image of the actual force acting at the tip is shown. It is evident that the force interactions varied in this case from $-174$ pN to $84$ pN. This resulted from the fact that the correction was performed linearly (see figure 11), whereas nonlinearities e.g. of the OBD contributed to the compensation inaccuracy as well (see figure 12(b)). As the protrusion structures exhibited a height of $20$ nm $\pm 2$ nm, the nonlinearity of the OBD detector additionally contributed to the recorded force variations. On the other hand, the maximum values were found near the scan field boundaries and on the edges of the TGQ-1 sample, which is related to the imperfections of the PID control loop, while on flat surfaces the error was not higher than $100$ pN.

In the third experiment the reference sample was scanned with the negative set point load force of $-1.40$ nN (see 13). Surface scanning with a negative load force is of great importance for the investigation of specimens sensitive to force loading such as microbiological cells, suspended nanowires and two-dimensional (2D) material membranes. Scanning with a negative force meant that instead of pushing the tip onto the sample surface, the PredPID controller overcompensated for the tip-sample attractive interactions resulting in a slight pull of the cantilever in the upwards direction. Adhesion forces caused the tip to remain in contact with the sample, so the presence of adequately strong adhesion forces is a must for imaging with negative contact force. During the scanning the load force changed in the range from $1.330$ nN up to $1.530$ nN whereas the biggest error occurred at the end of the protrusion edges. This corresponded with the limited speed of the PredPID controller, which was unable to respond fast enough while moving with a speed of two lines s$^{-1}$ over quite a tall structure.

In order to test the resolution of the NZ AFM technology the surface of a HOPG sample was observed; the results are shown in figure 14. The investigations at zero nN were performed in a scanfield of $8 \times 8$ $\mu$m$^2$. The load force varied in this case from $-219$ pN up to $120$ pN, as shown in figure 14(c). A cross-section verification was made using the surface topography and compensating Lorentz force images, figures 15(a) and (b). Two atomic steps of a height of ca. two nm were identified—see cursors 1 T and 2 T in figure 15(a). At the position of the atomic steps, a force of $20$ pN was measured, which was needed by the tip to overcome the atomic edges—see cursors 1 F and 2 F in figure 15(b). The increase in the force acting at the tip on the atomic steps was not linked to the PredPID control overshoot, as no such contrast was observed in the topography image (compare figures 15(a) and (b)).

Such behaviour is caused by the higher attractive forces acting between the tip and the sample surface at the atomic edges. In this area the open atomic bonds lead to a change in the interactions at the tip apex. Similar effects were observed in

![Figure 9. A force-distance plot with possible set point contact forces marked, recorded in NZ AFM mode.](image1)

![Figure 10. An NZ AFM image of a TGQ1 sample obtained with $+2.80$ nN contact force. (a) Vertical deflection (VD). (b) Control variable (CV). (c) Contact force (CF).](image2)
Figure 11. An NZ AFM image of a TGQ1 sample obtained with a zero nN contact force. (a) The vertical deflection (VD). (b) The control variable (CV). (c) The contact force (CF).

Figure 12. A measurement of the deflection linearity with bias current in the range from $-2.0$ mA up to $+2.0$ mA, measured with (a) a laser vibrometer and (b) an OBD deflection detector. The recorded characteristics translate to $3.2 \mu m$ of available deflection range, and a determined value of $\text{InvOLS} = 245 \text{ nm V}^{-1}$.

Figure 13. An NZ AFM image of a TGQ1 sample obtained with a $-1.40$ nN contact force (pull-off force cantilever operation regime). (a) The vertical deflection (VD). (b) The control variable (CV). (c) The contact force (CF).

Figure 14. An NZ AFM image of a HOPG surface at a zero nN contact force. (a) The vertical deflection (VD). (b) The control variable (CV). (c) The contact force (CF).
scanning tunnelling microscopy (STM) when the open atomic edges were observed and depending on the type of the crystal defects, a contrast in the current collected by the microscope tip was recorded [25]. In the NZ AFM technology these phenomena were followed by force interactions which were compensated for and measured (in other words determined in the quantitative way) by the Lorentz force induced in the active electromagnetic cantilever.

5. Conclusions

The results presented here confirm that NZ AFM is able to operate with a vertical resolution matching that of widely-available AFM systems. Furthermore, the procedure was presented for surface imaging in a zero force regime, where the attractive and the repulsive forces acting between the tip and the sample are compensated for. Imaging with a zero nN force is of interest in biological applications, where fragile samples are commonly used. This opens up the possible applications of NZ AFM for contact imaging of fragile samples using well-known forces. The ability to control the cantilever deflection using an integrated electromagnetic deflection actuator opens up new application possibilities for fast surface imaging and precise control of the load force acting at the tip. This is also vital for F-z surface measurements, in which the force interactions between the probe and the surface are analysed at various approach speeds of the probe towards the surface. The experiments performed also proved that FIB-machined diamond tips made it possible to image HOPG crystal edges with the expected contrast.

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Figure 15. A cross-section of the HOPG topography images. (a) The vertical deflection (VD). (b) The control variable (CV).
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