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Prototype of Fast SPM module for AFM experiments

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Contents

Executive Summary

This document describes the implementation of the FAST module, initially conceived for Scanning Tunnelling Microscopes, in a commercial Atomic Force Microscope (AFM) system, namely a Keysight Technologies 5500 AFM located at ICN2, to operate in fast scan mode in both contact and tapping operational modes. The technical description of the whole experimental set-up is given, including the interfacing of the AFM control electronics with the FAST module, the spectral characterization of the scanner, calibration of lateral dimensions, a description of the used cantilevers and samples, the optimization of the functional parameters such as feedback control and the modification of the control software to adapt to the AFM requirements. Examples are given involving both contact and dynamic (amplitude modulation) operational modes and finally, a short analysis of future implementations is provided. The experimental results show that frame rates above 8 frames per second (fps) in static (contact) mode and 1 fps in dynamic mode have been consistently achieved using commercial microfabricated silicon cantilevers with resonance frequencies below 1 MHz and silicon and PS-PMMA block copolymer reference samples with a pitch of about 38 nm. Thus, we can conclude that the FAST module can be implemented in commercial AFMs. However, a more user-friendly operation would require additional improvements of the control software.

1. Concept

The working principle of an AFM, where the deflection of a cantilever beam due to the attractive/repulsive interactions between a sharp tip and a surface is detected, has broadened the use of such instruments among the scientific community since they can be used in different environments (vacuum, air, gas and liquid) for a plethora of materials with differentiated physical properties (stiff, soft, conductive, insulating, etc.) covering applications in materials sciences, microelectronics, biology, metrology, etc. [1]. In the vast majority of cases, atomic resolution is not a requirement within the multidisciplinary AFM community and the interest is focused in the mesoscale, with nanometer-size features (with lateral dimensions typically above 10 nm) and sampling areas usually above 1 micron x 1 micron. Images acquired with sufficient resolution are obtained within several minutes, which makes their acquisition rather slow, e.g., an image with 256 lines scanned at 1 Hz (1 sec per line) takes more than 4 minutes to be completed. In addition, the surface and the tip can irreversibly evolve during image acquisition due to wear as well as due to inherent transformations due to e.g., temperature and humidity. Thus, there is a real need to reduce the acquisition time for practical purposes and also to open the possibility to study dynamic processes in real time and in operando.

Recent progress in high-speed AFMs has been achieved since the first report in 1991 [2] thanks to the development of ultrashort cantilevers [3], which exhibit higher resonance frequencies than those typically used in dynamic mode (commercially available cantilevers exhibit resonance frequencies in the 2-3 MHz range), and to the increase of mechanical and electronic bandwidths of the scanners, optical detectors and controllers, respectively [4-8]. Commercial AFM instruments designed to operate in fast scanning mode exhibit frame rates typically in the 10 fps range, as claimed by the manufacturers.

Here, we have extended the successful approach of the implementation of add-on units to commercial Scanning Tunneling Microscopes (STMs) in order to achieve video rate acquisition [9] to a commercial AFM. In the case of STM it has recently been shown that imaging at the milliseconds timescale can be performed, in this case exemplified by the adatom-promoted graphene growth on nickel surfaces at high temperatures, and thus the dynamics of surface processes can be explored in real time with atomic resolution [10].

2. Design specification

2.1 Experimental setup

2.1.1 Commercial AFM system used

Figure 1(a) shows an image of the commercial AFM used in the here reported JRA, a Keysight Technologies 5500 AFM (formerly Agilent 5500 and PicoPlus from Molecular Imaging) system installed at ICN2 and acquired in 2007. The AFM is housed in a homemade vibration/acoustic isolation system. Figure 1(b) shows the control PicoScan 3000 control electronics, the head electronics box and the AC mode controller (bottom left of the image) together with the dedicated PCs and additional instrumentation. The used Keysight N9447A breakout box can be observed at the center left of the image on top of a lockin amplifyer.

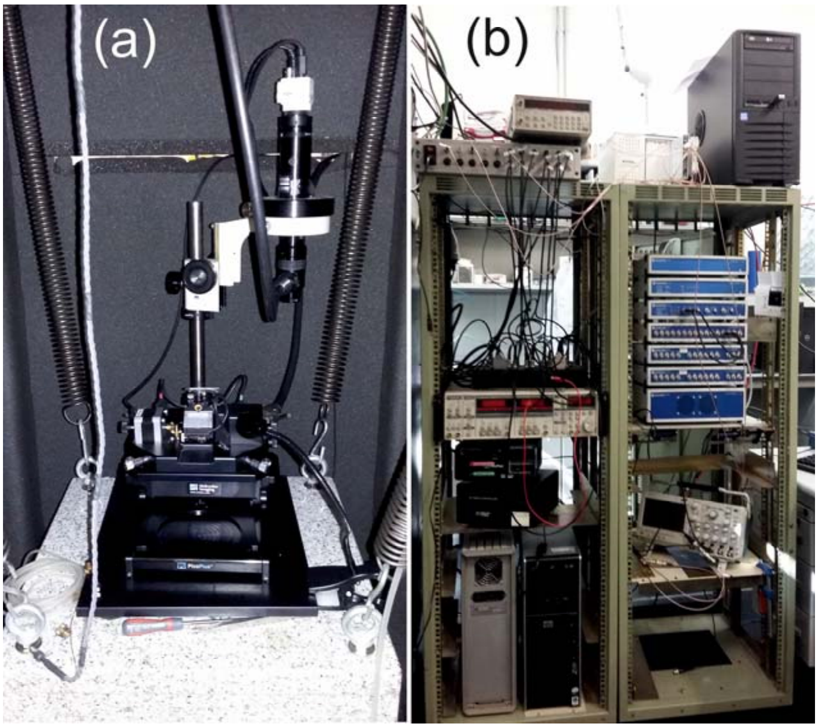

Figure 1: Image of Keysight Technologies 5500 AFM system for the implementation of the FAST module: (a) AFM housed in a vibration/acoustic isolation system and (b) control electronics and additional instrumentation.

We have used the Keysight N9524A multipurpose scanner (see Figure 2) which has nominal lateral and vertical scan ranges of 90μm and 7μm, respectively, for the available drive voltages of ±215V per axis that the controller electronics can deliver. The piezo actuator is mounted in a frame which holds the optical system including the laser and four quadrant photodetector (not shown in the picture) and a nose cone with a window conceived to work in liquid environment and the cantilever holder on top of the window. No modifications have been undertaken on the scanner for the operation in the FAST mode and has been used as for the standard (slow) mode.

Figure 2: Image of the N9524A scanner used in the set up

As compared to state-of-the-art instruments, the setup exhibits limited performance in terms of (i) photodetector bandwidth (<1 MHz), which hinders the use of cantilevers with resonance frequencies above 1 MHz, and (ii) the use of a laser spot that is too large for commercially available ultrashort cantilevers.

2.1.2 Integration of the FAST Module

The FAST module composed of both the APEX and the NI PXIe electronics can be seen on the upper part of Figure 1(b) and a scheme of the connections between the AFM controllers and the module is shown below in Figure 3 for both the contact mode (static) and dynamic mode configurations.

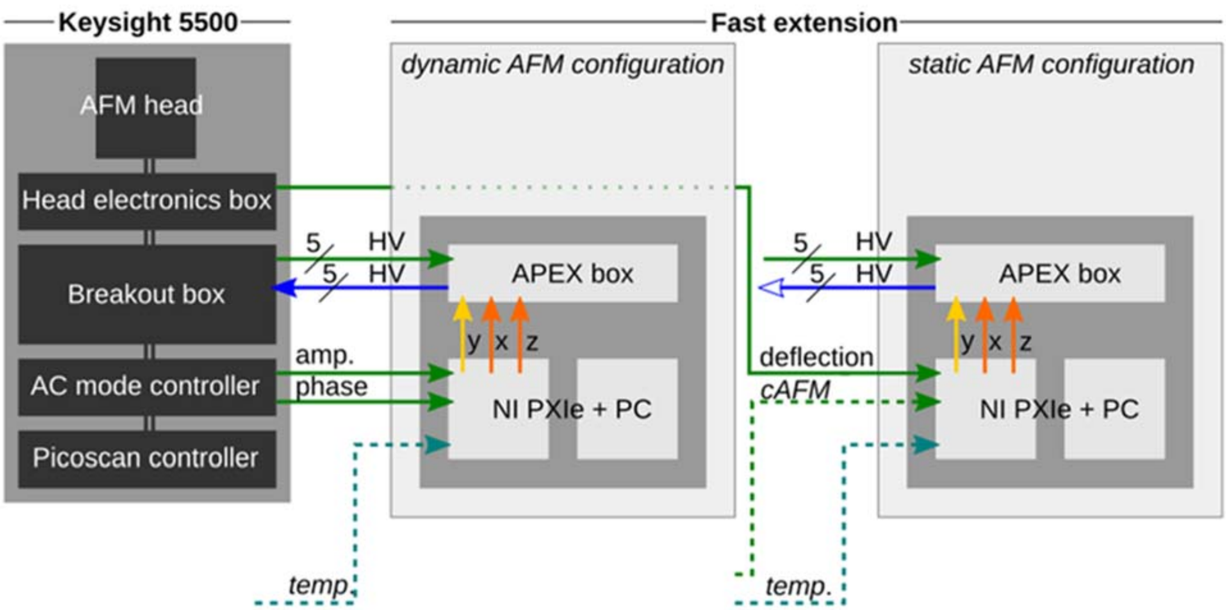

Figure 3: Scheme of the electrical connections between the AFM controllers and the FAST module.

The Keysight N9447A breakout box is used to exchange scanning signals between the FAST module and the host AFM system using standard 50Ω BNC coax connectors. The AFM high voltage (HV) positioning signals are branched out and fed into the FAST module, in which the fast scan signals are modulated onto the positioning "carrier" signal and then routed back to the AFM system in order to be applied to the piezoscanners, just as the carrier signal alone would do. The measurement variable used for the fast system depends on the chosen mode of AFM operation: (raw) deflection for contact mode AFM or amplitude and/or the phase signal for amplitude modulation dynamic (tapping) mode AFM, which are accessible via 50Ω BNC connectors located either on the Head Electronics Box or on the AC controller box of the AFM system. A passive RC 20dB attenuator is used to reduce the ±10V signal range of the AFM low voltage outputs to comply with the ±1V input range (2V pp in differential mode and an absolute maximum input ±2V, per connector) of the FPGA hardware (NI 5181R). In the setup arrangement one computer is dedicated to completing the AFM hardware and running the Keysight AFM software (PicoView 1.20.3 version) thus providing a complete conventional AFM system. A second PC serves the FAST extension alone, which houses the NI MXI-Express card to interface with the NI PIXe-1073 Chassis (which accommodates the FPGA card NI 5781R and DAQ card NI PXIe-6341) and is used to run the LabVIEW based control and acquisition software for the FAST module. External signals, such as temperature, can be also introduced for additional monitoring.

2.2 Technical description

2.2.1 Spectral characterization of the scanner

One of the critical characterizations before operation in the fast scan mode is the spectral response of the used scanner. This is indeed scanner and instrument dependent. Figure 4 shows the relative gain of the scan size in contact mode of the N9524A scanner up to 1kHz.

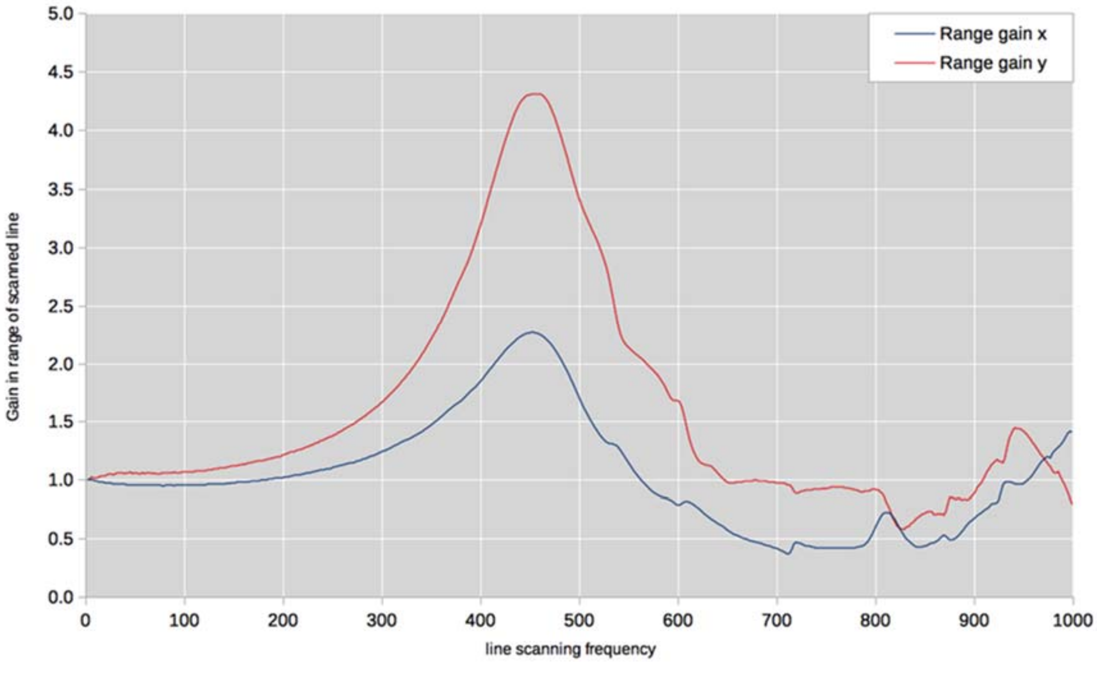

Figure 4: Spectral response of the N9524A scanner in the 0-1000 Hz range.

From the figure it becomes evident that the scan width is frequency-dependent [9] (the scan width depends on the piezo sensitivity of the actuator which is frequency dependent) and that beyond the resonance peak (at about 420 Hz) the gain drops below unity thus reducing the available range to levels below those at conventional scan rates. A systematic calibration of the absolute scan width as a function of the frequency is necessary but it may change with time at higher frequencies because the piezo may oscillate in demanding conditions (this will depend on the piezo material, dimensions, geometry and structure). For this reason we have calibrated the scan width with stable reference samples that exhibit well-defined patterns in the nanometer-scale range (see section 2.2.3). Special care has to be taken with wear, friction and cantilever torsion, in particular for soft cantilevers used in contact mode, since these may provide distorted images. When using soft samples friction accumulates material at the scan turning points which makes the determination of lateral dimensions quite cumbersome.

2.2.2 Cantilevers and samples used

We have used the following commercial cantilevers from Nanosensors:

<u>Contact mode</u>
PPP-CONTSCR (f_0=13kHz, k=0.2N/m, L=450um)

<u>Tapping mode</u>
PPP-NCHR (f_0=330kHz, k=42N/m, L=125um, W=30um)
qp-fast (f_0=800kHz, k=80N/m, L=40um, W=22um)

where f_0, k, L and W stand for the nominal resonance frequency, force constant, cantilever length and width, respectively. Since the main efforts have been dedicated to the implementation of the FAST module on the ICN2 AFM instrument we have not tried to make a comparison with cantilevers from other manufactures but and the selection has been based on our previous longstanding experience with the Nanosensors cantilevers.

For the fast operation mode we have chosen samples with fingerprint morphology and with a 38 nm pitch that have been used as internal lateral calibration references in the experiments [10]. The samples were fabricated at the Clean Room of the Institut de Microelectrònica de Barcelona (IMB-CNM) located close to ICN2. Two sets of samples were chosen: (i) lamellar PS-PMMA block copolymer thin films as received and after PMMA removal (see Figure 5) and (ii) silicon samples after transfer of the fingerprint pattern to silicon. Thus, the pattern is identical but the stiffness are clearly different (polymer vs. silicon). The as-prepared samples were first measured with a Bruker DIMENSION ICON instrument with Nanoscope V control electronics located at CNM with the piezoscanner calibrated using virtual standards [12].

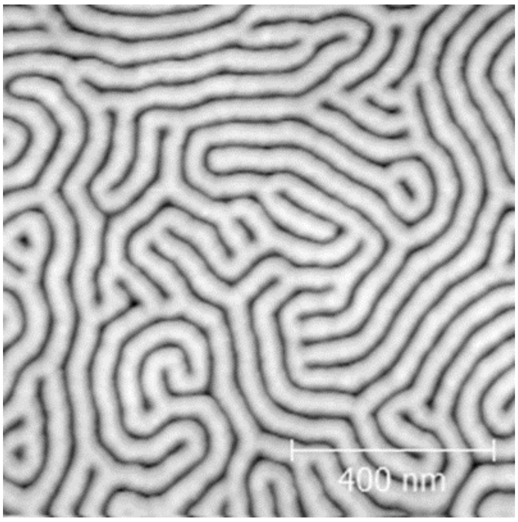

Figure 5: Tapping mode AFM image (conventional slow mode) of a PS-PMMA thin film after PMMA removal.

2.2.3 Feedback

The sample slope compensation is a key factor to consider for AFM since the target scan widths are noticeably larger than those used in STM under atomic resolution conditions. If such slope cannot be efficiently compensated by an accurate sample mounting and positioning and for samples with topography features in the low nm range, the feedback cannot be safely disabled, with the consequent penalty in terms of time acquisition. The FAST system does not provide a feedback loop for vertical position control of the scanned probe.

2.2.4 Software implementation

Considerable efforts have been devoted to the modification of the acquisition software in order to adapt it to the AFM operation (the software was initially developed for STM operation), find the optimum working parameters and extend the number of (fast) input channels from one to two. In the current hardware configuration, the number of fast input channels is limited to a maximum of two (bipolar). In the primary concept of fast AFM operation, the two input channels are dedicated to acquire the amplitude and phase signals of the cantilever oscillation (as provided by the Keysight hardware via BNC connectors). For tuning the feedback and fast axis linear slope correction settings, the amplitude signal is more helpful in assessing the quality of compensation, while the phase signals appear to provide better S/N contrast during imaging. However, the operator is free to use configurations to adapt to the variables of interest – as long as the specifications for the channel inputs, such as range and impedance, are obeyed. Hence for instance, in an alternative configuration one fast input channel could easily be repurposed to reading the cAFM preamp output or the amplitude/phase of higher order harmonics of cantilever oscillation as provided by a third party lock-in.

3. Results

Here we show selected results as a proof of the correct operation of the Keysight Technologies 5500 AFM in fast mode using the FAST module. Two examples corresponding to both contact and tapping mode are given as well as preliminary results on a study of the dynamics of the self-assembly process as a function of temperature. The experiments were performed as follows. After the sample was mounted on the sample stage and it was horizontally aligned manually with the adjustment knobs and after alignment of the optical system, images in either contact or tapping mode in the conventional slow mode were obtained in order to select a clean sample region avoiding protrusions and with a slope as small as possible. It is important to work in stable conditions and for this matter the several images were acquired until no drift could be observed. In absence of drift the slow mode was switched to the fast mode and the control parameters were optimized until the fingerprint structure could be clearly observed.

3.1 Contact mode

Figure 6 shows a topographic AFM image acquired in contact mode of the silicon sample in air taken at 8 fps. The area covered is about 600 nm x 600 nm, as estimated from the pattern pitch, under a moderate 100 pixels x 100 pixels resolution. The images have been acquired using PPP-CONTSCR cantilevers with nominal force constant of 0.2N/m and the line frequency of the fast scan axis is 800 Hz. As shown in Figure 4 this corresponds to a frequency region with a lower gain, so that the available scan width is lower as compared to the conventional low operation mode.

Above 8 fps the images become unstable although the fingerprint structure can be clearly identified. Thus, 8 fps seems to be a reasonable limit for fast acquisition for the particular scanner used. The integral (I) and proportional (P) gains of the feedback were I=0.5 and P=0, respectively.

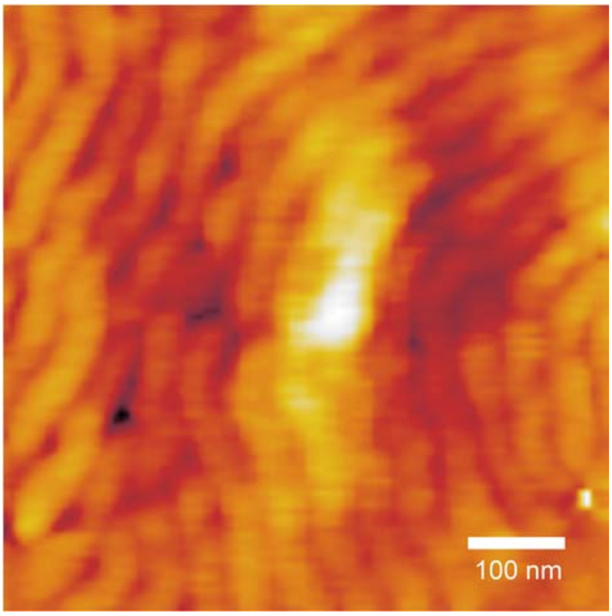

Figure 6: Frame from a FastAFM movie of a fingerprint-like silicon surface acquired in contact mode. The estimated scanned area is 600 nm x 600 nm and the line frequency of the fast scan axis is 800 Hz.

3.2 Tapping mode

Figure 7 shows a frame of a FastAFM movie obtained from the phase signal when scanning the PS-PMMA block copolymer sample in tapping mode using a qp-fast cantilever (Nanosensors) with a resonance frequency of 871 kHz and a nominal force constant of 80 N/m. The explored area corresponds to approximately 385 nm x 385 nm and was scanned at a frame rate of 1 Hz with a lateral resolution of 150 pixels x 150 pixels which corresponds to a line frequency of 150Hz.

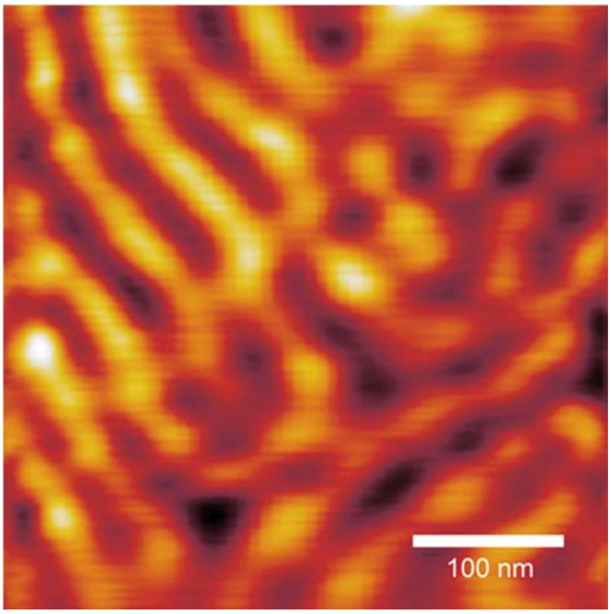

Figure 7: Frame from FastAFM movie of a fingerprint-like PS-PMMA sample acquired in tapping mode. The estimated scanned area is 385 nm x 385 nm and the line frequency of the fast scan axis is 150 Hz.

Because of the variation of piezo sensitivities with scanning rates (see Fig. 4), the periodicity of the fingerprint structures were used for lateral dimension calibration by comparing images acquired in fast scan mode with those obtained for the same surface in the corresponding standard AFM mode (i.e. "constant deflection/amplitude" feedback settings and line rate between 1 Hz and 2 Hz).

The integral (I) and proportional (P) gains of the feedback were I=0.008 and P=0 in tapping mode, respectively. In tapping mode, the trade-off of sampling frequency versus resonant frequency is crucial so that the number of oscillations per pixel is satisfactory. In our case, with a cantilever resonance frequency of 871 kHz, a line frequency of 150 Hz and 150 pixels, the estimated number of oscillations per pixel is 19.

3.3 Temperature dependent measurements

Towards the end of the activity, preliminary results were obtained in order to study the dynamics of self-assembly in block copolymers as a function of temperature. Unfortunately, the preparation of the heating stage in combination with the control electronics (331 temperature controller from LakeShore) took longer that foreseen because of technical problems. The runs were programmed as follows. A thin film of PS-PMMA on silicon wafers were prepared by spin coating close to the laboratory hosting the AFM and the as-prepared samples were mounted in the heating stage and then in the microscope. The idea was to acquire fast mode images as a function of temperature with the quest to observe the formation of the lamellae in real-time. Figure 8 shows a representative phase image of the initial formation of the self-assembly taken in conventional tapping mode. The unexpected formation of relatively high islands (about 25 nm high) prevents switching to the fast mode because of the need of feedback control and for this reason the measurement was performed in the conventional mode. The top image evidences dewetting of the film on the silicon substrate and the formation of lamellae at the borders of the islands. The image below shows a zoom of the structured regions. Further work should be performed along this line.

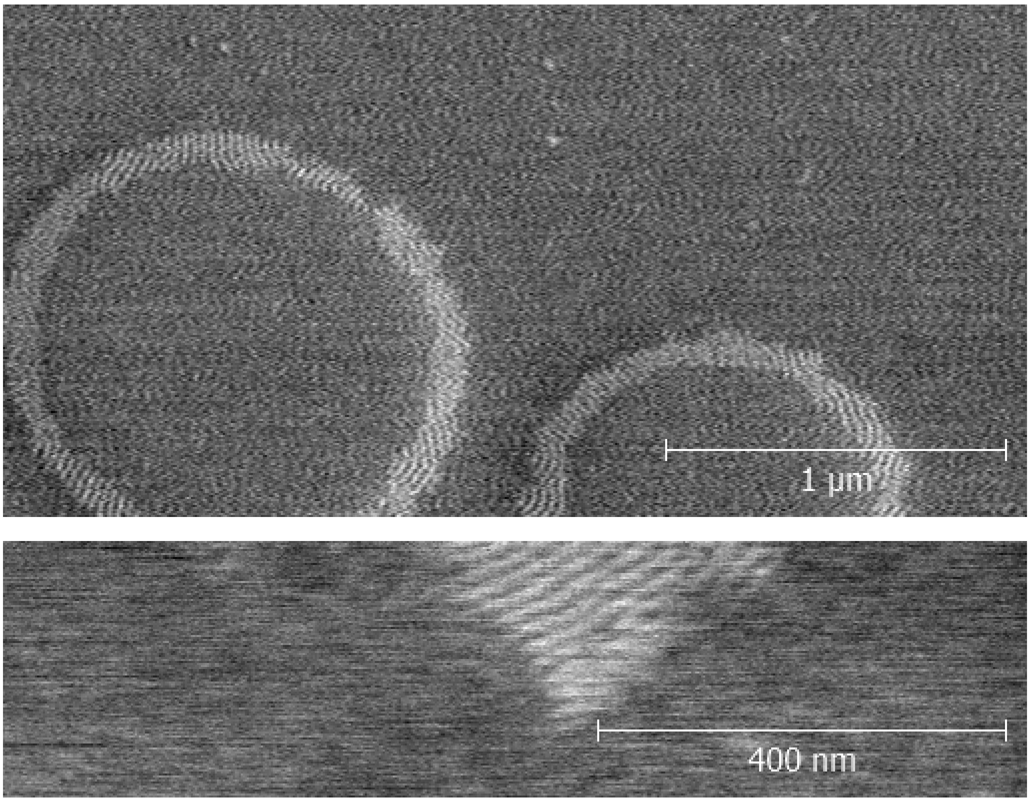

Figure 8: Phase images of a thin film of PS-PMMA prepared by spin coating on a silicon substrate and measured in tapping mode at 170 oC with Nanosensors PPP-NCH cantilevers (no reflective coating) with a resonance frequency of 287 kHz and at a scan speed of 2 lines per seconds.

4. Conclusions and perspectives

4.1 Conclusions

Fast scanning has been achieved with the ICN2 AFM using the FAST module with frame rates above 1 fps in spite of the physical limitations of the instrument, which was acquired in 2007. This instrument has been used as a test benchmark for other AFMs from different manufacturers and a list of relevant technical issues has been provided. After the optimization of the whole setup, switching from the conventional slow mode to the fast mode could be safely performed within 1 min by an experienced operator. However, this efficient and rapid switching can be only done when the samples are well mounted and aligned and show a smooth profile with corrugations typically below 5 nm, although this will depend on the actual tip radius. With AFM instruments exhibiting larger photodetector bandwidths and smaller laser spot sizes ultrashort cantilevers can be used which will enhance the fast scanning possibilities.

4.2 Perspectives

We include next a short list of suggestions that may be undertaken in the near future in order improve the integration of the FAST module in any kind of AFM, both commercials or home-made:

(i) Implement FAST module for control electronics such as the modular Nanonis SPM controllers with access to all signals via BNC connectors, a large bandwidth (5 MHz) and a LabView interface and which are used in several AFM instruments

(ii) Control software has to be improved in order to achieve a more user-friendly interface

(iii) Fast mode AFM measurements in order to investigate sufficiently slow real-time dynamics (within e.g., 10 pfs). Examples include self-assembly of block copolymers as a function of temperature, phase transitions and the effect of humidity on surfaces, among others.

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