Atomic force microscopy with time resolution of microseconds

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The atomic force microscope (AFM) can acquire high-resolution images under nondestructive conditions albeit at a very poor temporal resolution. This work presents two AFM mapping techniques, stroboscopic, and continuous, for imaging rapid periodical processes with nanometer spatial resolution and microsecond time resolution. Application of these methods is demonstrated for imaging very rapid cyclic changes in the position of a microfabricated grid. Motion was resolved with 10 nm spatial resolution, and 5 and 25 μs temporal resolution using the stroboscopic and continuous mode, respectively. The proposed methods have the potential to image wide variety of rapid processes with unparalleled combination of lateral and temporal resolutions. © 2005 American Institute of Physics. [DOI: 10.1063/1.1843277]

Traditionally (AFM) imaging has been restricted mostly to topological features due to poor temporal resolution. However, if the resolution of the AFM could be coupled with sufficiently high time resolution, this would present an important advance for imaging rapid structure dynamics in high resolution. Previous efforts to image motion with AFM have focused on rastering the cantilever over the sample as rapidly as possible. The time resolution of this rapid-scan technique is limited by the scanning speed and area. Presently, the time resolution of the rapid scan is on the order of a few tens of milliseconds at best. Much higher time resolutions are obtained when the AFM is used in the force sensing mode to detect vertical displacements from a single site.

We present methodology to obtain time-resolved AFM imaging based on the step-scan technique. In this method, an array of individual force sensing measurements is used to reconstruct topographic images. Similarly to the force sensing mode, the temporal resolution is limited only by the cantilever resonance frequency and the data acquisition electronics, while being independent of the scanned area. The setup consisted of a commercial AFM equipped with a closed-loop scanner. To drive the cantilever in a stepwise manner, dc voltage offset was applied to either X or Y piezoscanner. In our experimental setup, an electrostatically actuated piezo, with attached calibration grid, is oscillated at 5 kHz. At each point, the vertical motion is recorded directly from the PSD while the cantilever is at rest for a duration set by the required signal-to-noise ratio. The PSD data are fed into the oscilloscope and are averaged using the same triggering signal that actuates the piezo/calibration grid sample to improve signal-to-noise ratio. The averaged trace for the pixel is stored, the cantilever moves to the next pixel, and the process repeats. At the conclusion of the scan, a trace of the ac motion of each pixel is obtained. In the stroboscopic method, the system returns to its resting state before the probe is moved to the following pixel, ensuring that each pixel’s motion trace begins from the resting state. The dc value of each pixel (obtained from the dc image) is then combined with its ac motion to reconstruct a complete time lapse image of the sample, including both motion and surface features.

Two methods are introduced to achieve time-resolved imaging: the stroboscopic and the continuous time-resolved AFM. Both methods are based on imaging periodic processes, with the ability to obtain the motion of each point on the sample individually. The movement of the entire sample is reconstructed by arranging the time-lapse images of each point together according to their spatial location.

In the stroboscopic method, the z-piezo and PSD data are collected directly from the AFM. Initially, the AFM cantilever is moved in a stepwise fashion along a virtual matrix overlaying the motionless sample. The voltage data from the z-piezo (termed the dc value) are taken at each point, and a stationary topographic image, denoted as the dc image is reconstructed. Next, the motion of the sample is continuously triggered (i.e., strobed) by an external source, and the cantilever is stepped backwards over the same virtual grid. In our experimental setup, an electrostatically actuated piezo, with attached calibration grid, is oscillated at 5 kHz. At each point, the vertical motion is recorded directly from the PSD and the cantilever is at rest for a duration set by the required signal-to-noise ratio. The PSD data are fed into the oscilloscope and are averaged using the same triggering signal that actuates the piezo/calibration grid sample to improve signal-to-noise ratio. The averaged trace for the pixel is stored, the cantilever moves to the next pixel, and the process repeats. At the conclusion of the scan, a trace of the ac motion of each pixel is obtained. In the stroboscopic method, the system returns to its resting state before the probe is moved to the following pixel, ensuring that each pixel’s motion trace begins from the resting state. The dc value of each pixel (obtained from the dc image) is then combined with its ac motion to reconstruct a complete time lapse image of the sample, including both motion and surface features. With the motion of each pixel recorded and the knowledge that all pixels are in phase with each other, arraying all of the time-lapse images together and reconstructing an image of the motion of the entire sample is straightforward. Figure 1 shows a snapshot taken from the time-lapse movie showing the surface topographic image after 500 ms.

Although the motion of many interesting systems can be repetitively triggered after each cycle, for others it is technically challenging or impossible. In order to extend our method to a wider variety of systems, we present a continuous time-resolved AFM methodology. In this method, data are continuously gathered, thus eliminating the requirement for resetting the system after each cycle (i.e., strobing). As in...
the stroboscopic method, the data stream recorded in the continuous mode. (a) and (b) show the voltages corresponding to the x and y position of the cantilever, respectively. Each voltage step corresponds to 10 nm, and location on the grid can be determined from the voltage levels of the A and B traces. The z-piezo and PSD signal are shown in (c) and (d), respectively. All four channels of timestamped data are streamed continuously and stored, as the cantilever moves over a virtual grid overlaying the sample. At each transition (marked by arrows), the entire data stream is cut and each segment of data corresponds to the motion of a particular point on the sample. The z-piezo signal contains slow motions and the dc characteristics of the sample, while the PSD shows the rapid motions.

FIG. 3. (Color online) The motion traces of all pixels plotted in contour maps. Two cycles of motion, spanning 2 ms are shown. (a) Traces before phase alignment; (b) traces after the course alignment process. A phase error that is linear with acquisition time remains. The maximum phase error occurs at the last pixel and is 5.5%; (c) traces after application of the frame matching algorithm during the refinement process. The phase error is largely eliminated and the remaining error has a rms value of 0.19%.

FIG. 2. (Color online) Four seconds representative sample of the data stream recorded in the continuous mode. (a) and (b) show the voltages corresponding to the x and y position of the cantilever, respectively. Each voltage step corresponds to 10 nm, and location on the grid can be determined from the voltage levels of the A and B traces. The z-piezo and PSD signal are shown in (c) and (d), respectively. All four channels of timestamped data are streamed continuously and stored, as the cantilever moves over a virtual grid overlaying the sample. At each transition (marked by arrows), the entire data stream is cut and each segment of data corresponds to the motion of a particular point on the sample. The z-piezo signal contains slow motions and the dc characteristics of the sample, while the PSD shows the rapid motions.

FIG. 1. (Color online) A representative frame from the time-lapse movie of the sample motion measured with the stroboscopic time-resolved AFM method. The image is reconstructed from 484 pixels (22×22) which are 10 nm apart. Overall the image covers an area of 220×220 nm². For the time-lapse movie, see EPAPS. Ref. 7.
fundamental periods are in phase with each other. We therefore identify the earliest time point in each pixel trace that is separated from the first pixel’s start time by an integral number of fundamental periods. The start of each pixel trace is shifted to those time points. The first stage of the phase alignment described above highly depends on the accuracy of determining the fundamental frequency (and consequently the fundamental period). An error in the fundamental frequency ($\Delta f_0$) causes an error in phase proportional to the elapsed time

$$\left( \frac{\Delta f_0}{f_0} \times t_e \right),$$

where $t_e$ is the elapsed data acquisition time. This phase error becomes significant for large numbers of pixels [Fig. 3(b)], and appears as a traveling wave artifact superimposed over the motion of the sample. To eliminate the phase error we applied the frame matching algorithm in the refinement stage. The frame in the reconstructed motion that corresponds to the dc image is selected, and a complementary phase shift proportional to each pixel is introduced. The phase shift is optimized by minimizing the least-squares difference between the selected frame and dc frame. With the phase error known, each pixels motion trace can be appropriately adjusted [Fig. 3(c)], and a 3D matrix representing the samples motion as a function of time is constructed as in the stroboscopic method.

In conclusion, the step-scan time resolved AFM opens the door for imaging rapid periodic motion of various biological systems as well as nonbiological systems. We demonstrated that the technique can visualize motions of micro-electromechanical systems (MEMS). Future progress in nanotechnology is likely to produce nanomotor devices, whose structures’ performances could be directly imaged with this technique. We anticipate, however, that step-scan time resolved AFM will have the most significant impact on studies of biological systems. We have the potential to image rapid periodic biological processes as they occur in real time under physiological conditions with an unprecedented combination of lateral and temporal resolutions.

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7 See EPAPS Document No. E-APPLAB-86-018501 for a time-lapse movie. A direct link to this document may be found in the online article’s HTML reference section. The document may also be reached via the EPAPS homepage (http://www.aip.org/pubservs/epaps.html) or from ftp.aip.org in the directory /epaps/. See the EPAPS homepage for more information.