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Circular mode: A new scanning probe microscopy method for investigating surface properties at constant and continuous scanning velocities

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In this paper, we introduce a novel scanning probe microscopy mode, called the circular mode, which offers expanded capabilities for surface investigations especially for measuring physical properties that require high scanning velocities and/or continuous displacement with no rest periods. To achieve these specific conditions, we have implemented a circular horizontal displacement of the probe relative to the sample plane. Thus the relative probe displacement follows a circular path rather than the conventional back and forth linear one. The circular mode offers advantages such as high and constant scanning velocities, the possibility to be combined with other classical operating modes, and a simpler calibration method of the actuators generating the relative displacement. As application examples of this mode, we report its ability to (1) investigate the influence of scanning velocity on adhesion forces, (2) measure easily and instantly the friction coefficient, and (3) generate wear tracks very rapidly for tribological investigations. © *2011 American Institute of Physics*. [doi:10.1063/1.3658049]

I. INTRODUCTION

The advent of scanning probe microscopes (SPMs) and especially of the atomic force microscope (AFM) have opened new perspectives for investigating at the nanoscale phenomenological mechanisms that are encountered in different fields such as material science, biology, tribology, thin films, microelectronics,[1,2] etc. The SPM principle is based on measuring interaction forces between the probe and a sample. Conventionally, the SPM is employed for imaging surface topography with high resolution either in dynamic modes[3,4] or contact modes.[5] For this application, the scanning motion of the probe relative to the plane of the surface is a back and forth displacement with amplitudes ranging from 10 nm up to 100 μm and frequencies usually ranging from 0.1 up to 10 Hz. It also offers the capability of measuring probe-sample interactions as a function of their separation distance (force spectrum).[6] Finally, it has evolved into a versatile instrument for measuring physical properties such as friction,[7] resistivity, capacitance,[8] etc.

Despite the various modifications that have been introduced to the basic SPM setup for expanding and improving its measurement capabilities, there still remain multiple challenges that hinder the accuracy of the experimental procedures. As a main limitation, the conventional back and forth scanning motion of the probe that results in rest periods when the direction of the scan is inverted. During these rest periods, the characteristics of the nanoscale probe-sample interaction may change significantly. For example, in the case of hydrophilic surfaces, the probe-sample contact may evolve due to the formation of a water meniscus when the contact is at rest for a few milliseconds.[9] This leads to a capillary force that acts as an additional normal load.[10] Consequently, the resulting adhesion forces induced by the evolving meniscus are never constant and the average value of the adhesion force depends on experimental conditions (scanning length and frequency, etc.). The scan inversion may also change significantly the probe-sample interactions such as shear stress. As a consequence, while experimenting under conventional scanning conditions, the measurements of the probe-sample interactions are always conducted in a non-stationary state.

To address this problem, we present an innovative SPM setup that allows achieving a circular displacement of the probe relative to the sample rather than a back and forth linear displacement. This new SPM mode is called the circular mode.[11] This original mode offers significant advantages such as: (1) allowing collection of data at constant and continuous velocities without any halt during the entire scan duration (if no stick-slip effect is considered) and thus to attain stationary state and (2) reaching high scanning velocities higher than 100 000 μm s^{-1}. Such advantages are determining in physics for quantitative measurements at a local scale that require high-speed displacements or if the probe-sample interaction could be influenced by rest periods, acceleration, deceleration, or non-constant velocities. In the following, we will present the experimental setup of the circular mode. Then, we will report some experimental data that demonstrate the scientific potential of this new mode in a wide variety of applications.

II. CIRCULAR MODE: EXPERIMENTAL SETUP

The SPM is dependent of three components: the most sensitive component which is the probe that interacts directly with the sample surface, the photo detector that senses the changes in the angle of the reflected beam due to the

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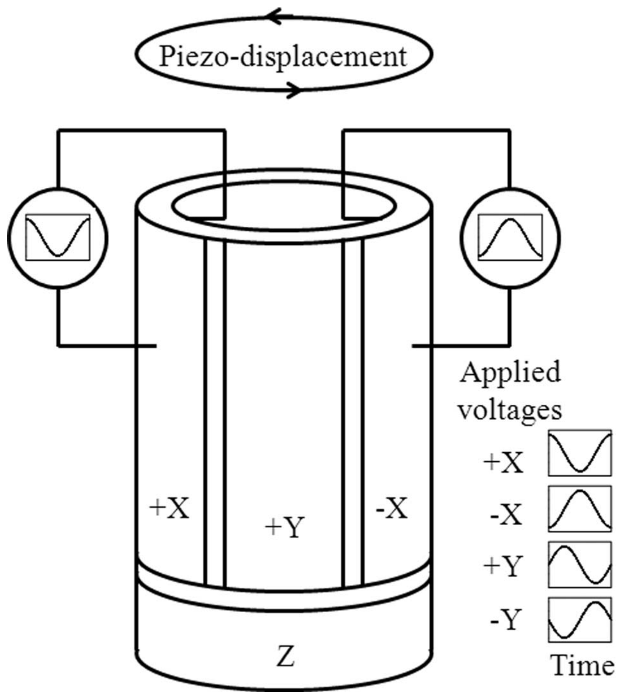

FIG. 1. Applied voltages to the piezo-actuator tube as a function of time to generate a circular displacement. A cosine and a sine voltage at the same frequency are applied to the X and Y electrodes, respectively. The opposite voltages are applied to the −X and −Y electrodes.

deflection of the cantilever following the tip-sample interaction, and the three-dimensional (3D) scanner that facilitates accurate and precise relative displacements of the probe. Two scanning configurations are available: scanned tip SPM where the piezoelectric scanner is rigidly attached to the probe and is moved over the sample surface which stands still, or scanned sample SPM where the scanner is attached to the sample and it is moved under the motionless tip. The SPM scanner, which is the core unit of the experimental setup, consists of piezoelectric materials, usually ceramic actuators. The application of a voltage on the inner and outer surfaces of the ceramics causes their length to increase or decrease, depending on the polarity of the voltage applied. The scanner configuration consists of combining independently five electrodes in a hollow piezoelectric tube (Fig. 1) that are responsible for the movement along three axis X, Y, or Z, where X and Y define the horizontal plan axes and Z defines the vertical axis. For achieving a horizontal displacement in the X direction, the two opposite X and −X electrodes are fed by an opposite voltage that causes the piezo-actuator to bend, whereas for achieving a vertical displacement, it is sufficient to apply voltage to the Z electrode that causes the piezo-actuator to extend or retract.

Generating a relative circular displacement of the probe-sample contact necessitates a particular control of the piezoelectric ceramic motion. Two sinusoidal voltage signals phase shifted by $\pi/2$ must be applied to the electrodes that refer to the axes defining the plane of the sample surface [−X, X] and [−Y, Y] (Fig. 1). A digital-to-analog converter (DAC) device, or a lock in amplifier or a sinusoidal tension generator combined with a dephasor can be used to generate the voltage signals. A SPM signal access module (SAM box from Veeco, USA) allows access to internal SPM signals and is used to apply the following signals to the ceramic actuators:

$$\mathrm{V_X} = \mathrm{V}\cos\omega t, \tag{1}$$

$$\mathrm{V_Y} = \mathrm{V}\sin\omega t, \tag{2}$$

$$\mathrm{V_{-X}} = -\mathrm{V_X}, \tag{3}$$

$$\mathrm{V_{-Y}} = -\mathrm{V_Y}, \tag{4}$$

where V is the voltage half-amplitude and ω is the angular frequency.

The circular mode offers two parameters of control to regulate the velocity of the probes displacement relative to the surface. The voltage half-amplitude V, which sets the radius R of the circular movement, and the voltage angular frequency ω, which defines the interval of time, needed to make a complete circle. Thus, the scanning velocity v is given by the following equation: $\mathrm{v} = \mathrm{R}\omega$.

Finally, it is worth mentioning that the circular displacement could be easily implemented in a commercial SPM since one has access to the internal signals of the piezo-actuator. Technically, the commercial SPMs just require a minor software modification for implementing the circular mode. Moreover, the circular mode could be either employed with an open-loop or a closed-loop SPM system. However, the implementation of the circular mode on a closed loop SPM system has not been tested.

III. ADVANTAGES OF THE CIRCULAR MODE

The circular mode offers advantages that are essential for determining surface properties at the nanoscale. It offers the possibility to operate at high scanning velocities for analysing smooth surfaces as no servo-loop is required to maintain a quasi-constant load. For our SPM (Dimension 3100, Nanoscope V from Veeco, USA), the horizontal resonance frequency of the piezo-actuator (called G scanner) is about 450 Hz. Considering a full-scale scan of 110 μm, it is technically possible to generate scanning velocities higher than 100 000 $\mu\mathrm{m\,s^{-1}}$. These velocities are typically three orders of magnitude higher than the ones obtained with a conventional SPM and they are comparable to realistic velocities generated in macro-, micro-, or nano-devices. Collecting data at these realistic velocities offers new opportunities for investigating velocity dependent properties of friction, adhesion or wear. Consequently, it is possible to conduct measurements with high resolution and more realistic conditions.

However, it is not certain if operating under these conditions (high displacements at high frequencies) may or may not damage the piezo-actuator. In our particular operating conditions, the voltage half-amplitude and the frequency are limited to 10 V and 100 Hz leading to a displacement velocity of about 1200 $\mu\mathrm{m\,s^{-1}}$.

A further benefit of the circular mode is the continuous and constant scanning motion which allows avoiding inconveniences caused by the rest periods of the probe resulting from the abrupt inversion of the scanning motion encountered when using the conventional back and forth scan mode.

Moreover, for an open-loop SPM system, the circular mode needs a far less complicated method for calibrating the piezo-actuator displacement. Indeed, in classical SPM scanning, back and forth scans are realized in the so-called fast scan direction. These consecutive scans are slightly shifted perpendicular to the fast scan direction (called the slow scan direction) and are added one by one to form the image. Because of the nonlinearity and creep of the piezo-actuator, the voltages applied on the two directions of the piezo-actuator follow a complex equation that requires the calibration of three parameters to generate a voltage function that conducts to a linear scan.[12] In particular, the G scanner (Veeco, USA) requires the calibration of 14 parameters that are determined through a relatively long and complex calibration process. However, when using the circular mode, there are no more fast and low scanning directions. The voltage sent to the piezo-actuator is sinusoidal with respect to time for the two horizontal directions. Thus, one should only consider the nonlinearity of the scanner sensitivity for each direction leading to a simpler and reliable piezo-actuator calibration. Practically, the calibration of the piezo-actuator for obtaining an accurate circular motion of the probe can be easily realised using three different methods: (1) The classical calibration method that requires the use of a reference sample such as a calibration grid. In this case, the authors propose to impose sinusoidal voltages of various amplitudes in either the X or Y direction and to measure the resulting displacement. This method is mostly adapted for low frequency displacements as an effective servo-loop is required to generate the "height" signal. (2) The circular displacement could be calibrated with the displacement sensors of a closed-loop SPM if their working frequencies are compatible with the frequency of the circular displacement. (3) The last method consists in measuring the dimension of a circular track due to wear or plastic deformation obtained with the circular mode at a fixed amplitude and frequency. This is particularly interesting as it is simple, fast, and reliable even if it is less accurate than the conventional method due to the error source on the width of the track. This method is especially adapted when the piezo-actuator is used at high frequencies since the servo-loop is generally not efficient at these frequencies. Figure 2 clearly shows the evidence of a circular track due to wear generated by the circular motion of the tip-sample contact. Obviously, this method is damaging for both the sample and the probe.

Finally, another interesting feature of the circular mode is that along with the possibility of combining the circular mode to the classical modes, as adhesion force mode or friction force mode. Consequently, it is possible to measure simultaneously adhesion forces or friction forces while the relative probe-sample displacement is circular. Such advantages of the circular mode can be essential for metrological applications. The main characteristics of the circular mode are summarized in Table I.

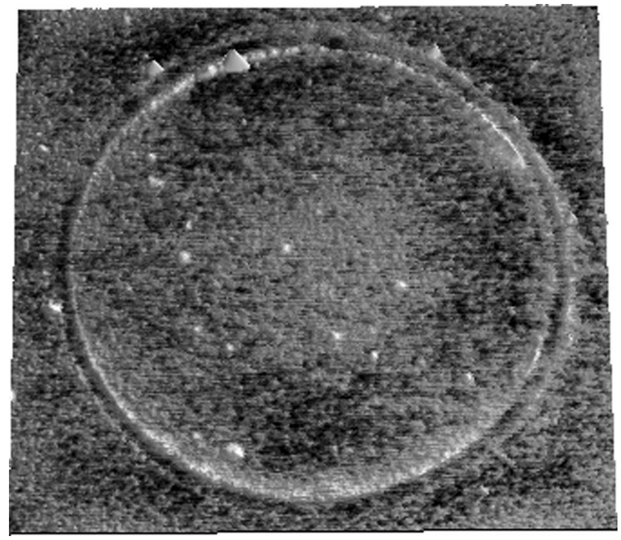

FIG. 2. AFM topographic image of a GaAs thin film surface (image size: 3 μm $\times$ 3 μm). The circular track is created by the circular displacement of the probe relative to the sample in using the circular mode at a sliding velocity of 1000 μm s^{-1} for three consecutive minutes, under a load of 80 nN.

IV. APPLICATION EXAMPLES OF THE CIRCULAR MODE IN NANOTRIBOLOGY

A. Coupling the circular mode with other mode: Measurements of adhesion forces at different sliding velocities

Conventional AFM force mode allows the measuring of the adhesion force by means of a force spectrum.[6,10] The force spectrum is obtained by imposing back and forth vertical displacements to the piezo-actuator. It is possible to combine the circular mode with the conventional force spectrum. In such case, one can acquire force spectrum while the probe is scanning in a circular motion at a fixed velocity. This is advantageous for investigating the sliding velocity dependence of the attractive and adhesive forces. Force-distance measurements combined to the circular mode were conducted on various hydrophilic surfaces (mica, silicon nitride, silicon wafer, chemical vapour deposited (CVD) gold surfaces) at a relative humidity close to 40% in air. Figure 3 shows the force-distance spectrum obtained on gold for four sliding velocities. The four velocities are obtained by imposing a circular displacement at a frequency of 100 Hz with a diameter of 0, 0.32, 0.95, and 3.2 μm, respectively. The corresponding applied voltage is computed based on the calibration of the piezo scanner using the wear track method described previously. Voltage signals applied to the piezo-actuators were generated by a homemade software and a DAC card and sent to the 3D scanner via the SAM box (Veeco, USA). The vertical displacement was set to 500 nm at a frequency of 0.1 Hz corresponding to a vertical velocity of 0.1 μm s^{-1}. The adhesion forces corresponding to the jump-off cantilever deflection (Fig. 3) is decreasing with an increase of the sliding velocities. At high sliding velocities, the adhesion force reaches a minimum value that remains constant and equal to the attractive force.[13] To explain such a behaviour, it is well known that the capillary force between the probe and the sample

TABLE I. Main differences and similarities between circular and back and forth scanning mode.

Item	Circular mode	Back and forth mode
Actuating the scanner	In both directions: sinusoidal voltages, no harmonics	In the fast scanning frequency quasi triangle voltage, many harmonics
	Easy to achieve constant velocities whatever the frequency	Difficult to achieve displacements at constant velocities especially at high frequencies
	Fast calibration process	Time-consuming calibration process
	Smooth and constant scanning	Strong inversion of scanning direction two times a line
	No stop periods (excepted stick-slip), accelerations and decelerations	Stop periods, accelerations and decelerations occur at inversion
	Easily achievable stationary state	Unachievable stationary state due to stop periods and inversions of the motion direction
Coupling with other modes	Both modes could be	
	Combined with LFM mode, but back and forth conventional mode allows acquiring friction loops to achieve local friction measurements, whereas circular mode allows measuring friction force at a constant and continuous sliding velocity (servo-loop should be inactive)	
	Combined with force spectrum but this combination is not implemented in commercial AFM	
	Combined with others SPM modes: STM, SNOM, contact AFM, Tapping mode, etc.	

plays at the nanometer scale, a significant role for hydrophilic surfaces and at humidities higher than 30%.[14,15] When performing the circular sliding of the probe on the sample, the capillary force is affected by the increase of the velocity leading to a decrease of the adhesion force (Fig. 3). We conclude that the capillary meniscus vanishes at high sliding velocity as previously suggested.[16–18] This assumption is confirmed by adhesion force measurements conducted under argon (no capillary meniscus can form), which show comparable adhesion force values obtained from force curves measured with the circular mode in air at high sliding velocity on different surfaces such as silicon wafer and CVD gold surfaces.

Thus, the circular mode can be used to overcome the problems related to the capillary condensation for (1) reducing the applied load by eliminating the capillary forces and (2) measuring interaction forces with or without capillary forces without additional equipment.

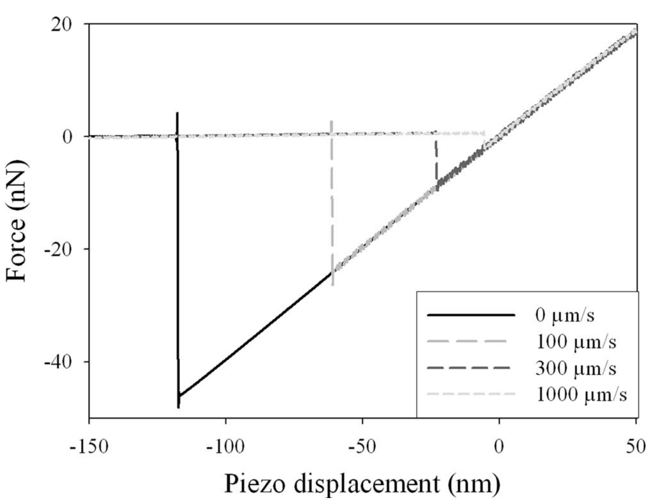

FIG. 3. Force spectra acquired on a CVD gold layer at four different sliding velocities. As the sliding velocity increases, the adhesion force decreases.

B. Measurements of friction forces at different loads in a stationary state: Instantaneous determination of a friction coefficient

In classical friction measurement the probe is scanned back and forth (Y axis direction) perpendicular to the main axe of the cantilever (X axis direction). The friction force induces a torsional response of the cantilever that is measured by the lateral force microscopy (LFM) signal and the friction force values are computed from the friction loop, i.e., the difference between the LFM signal of the back and the forth displacement.[19] When performing a circular motion, the approach is different. The friction force should be decomposed in two terms, the parallel F_X and perpendicular F_Y components of the friction force. Consecutively to the circular motion, the perpendicular Y component of the friction force generates an alternative torsion of the cantilever and the LFM signal has a sinusoidal shape for which the amplitude is proportional to the friction force. Similarly, the parallel X component of the friction force generates an alternative bending of the cantilever and the "error" signal has a sinusoidal shape. If the servo-loop is active, this will generate a sinusoidal displacement of the piezo-actuator in the Z axis direction leading to a non constant load and a coupling between normal and friction forces. If the servo-loop is not active, the LFM and "error" signals have a sinusoidal shape but there is no coupling between the two signals. Thus, in our experimental conditions, according to the above comments, the local friction force cannot be measured accurately, as it is measured in the case of a back and forth conventional scan, because (1) the sliding direction only occurs parallel to the X axis direction, twice in a single turn; (2) the friction loop cannot be realised as the probe is always turning in the same way; and (3) if the servo-loop is active, there is a coupling between friction and normal forces.

Nevertheless, if one assumes that the friction force does not depend much on the location; it is possible to measure an average friction force by measuring the amplitude of the LFM

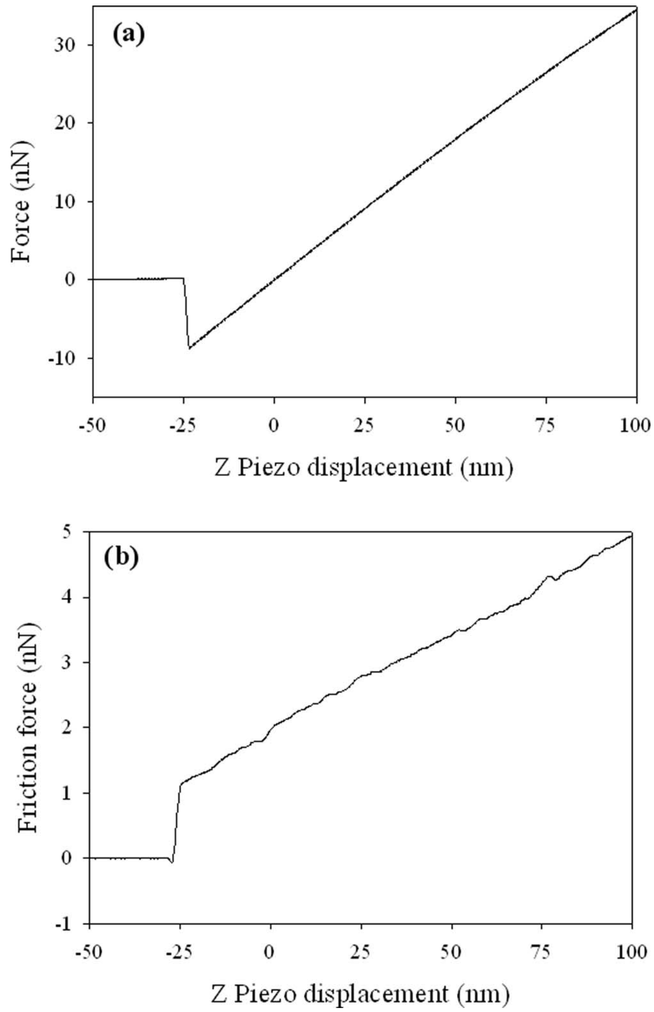

FIG. 4. Adhesion and friction spectra conducted on a gold sample at a sliding velocity of 300 μm s^{-1}, show simultaneous acquisition of the normal (a) and friction force (b) as a function of the piezo-actuator displacement. The graph allows direct computation of the friction-load dependence.

signal during circular motion and without using the servo-loop. Previous authors have suggested similar method using lateral force modulation[20–22] but the circular mode offers the advantage of reaching high, constant and continuous sliding velocities and thus stationary states. A lock-in-amplifier enables measuring the amplitude of the LFM signal at the frequency of the circular movement and thus measuring directly the friction force. In combining the circular and force spectrum mode, it is therefore possible to obtain simultaneously adhesion and friction-load spectra (Fig. 4). One can rapidly and easily acquire the friction-load dependent curve, which requires time-consuming experiments with the conventional AFM mode.

Concerning the calibration of the lateral force, it could be carried out using the classical methods developed for calibrating the LFM signal using the back and forth method since lateral forces in circular mode are also measured from the torsion of the cantilever. For these experiments, the lateral force signal has been calibrated using the method proposed by Ogletree *et al.*[19]

In our experiments, the Amontons law, which predicts a linear dependence between friction and load, is verified. The slope of the friction force versus the load curve gives directly the value of the friction coefficient (Fig. 4(b)). In this example, we obtain a fiction coefficient of 0.085 for a gold CVD layer and a sliding velocity of 300 μm s^{-1}.

C. Using the circular mode at high sliding velocity: Fast achievement of wear tracks

Wear can be defined as a process in which interactions of the surfaces or bounding faces of a solid with its working environment results in dimensional loss of the solid. A serious issue is that wear rate is known to depend strongly on the magnitude of the loading force, leading to a dramatic variation of the wear rate as the sliding conditions change. However, for different operating conditions (materials, geometry, roughness, humidity, etc.) and sliding velocities, friction and wear are dominated by different mechanisms.[2,7,23] Furthermore, wear at the nanometer scale is generally a slow process that results in low depth wear tracks that are difficult to measure. The circular mode helps to investigate the evidence of

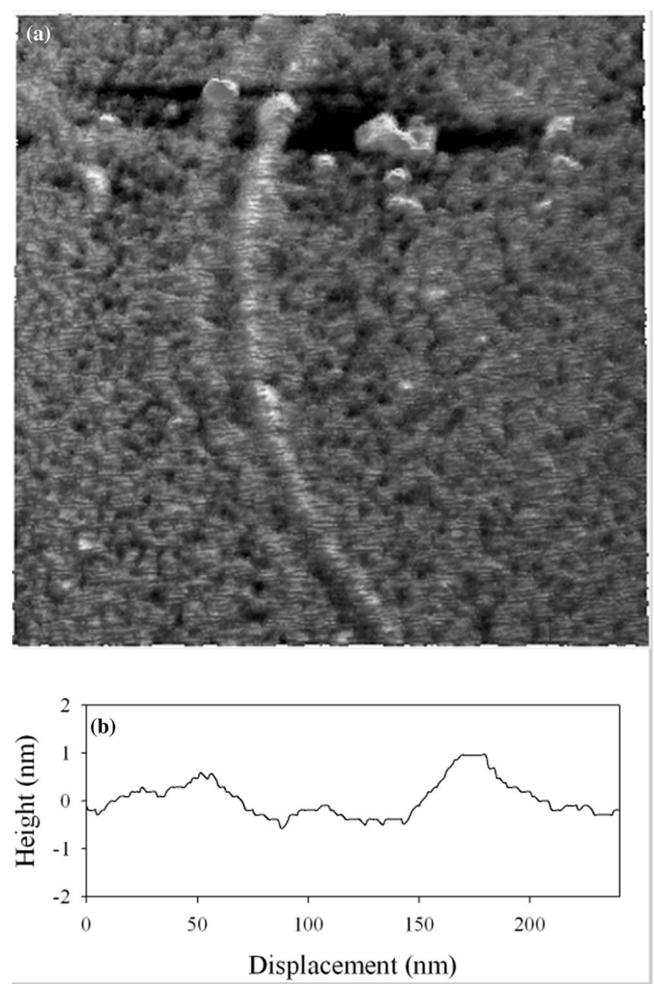

FIG. 5. Wear track obtained with the circular mode on a GaAs, sample (a) magnified image of the track generated by the circular motion for 3 consecutive minutes, under a load of 80 nN at a sliding velocity of 1000 μm s^{-1}. (b) Profile showing the depth of the track.

wear by increasing the sliding velocity and thus generating a faster wear process. For example, an experiment conducted on a gallium arsenide (GaAs) sample with a silicon nitride probe, at a load of 80 nN and at a sliding velocity of 1000 $\mu m\,s^{-1}$ requires 3 min to generate a track, which is about 0.5 nm in depth (Fig. 5). The equivalent experiment conducted with a conventional AFM needs about thirty minutes. This duration is generally too long to prevent any drift of the piezo-actuator that disturbs the measurements.

The circular mode could be employed for tribological investigations as, for example, understanding the mechanisms implicated in wear or characterizing wear properties of materials. In a more general way, the circular mode could be employed for changing rapidly the surface properties that can be modified by the probe-sample interactions. As examples, (1) circular features generated by wear tracks, lithography, oxidation, or any modifications of the physical surface properties (magnetic, electrostatic, etc.) could be employed for data storage and (2) the circular mode could be employed to polish surfaces or for machining surfaces by wear, by combining the circular mode with a translation displacement. It is possible to generate easily and rapidly surface features of any form by this process by using abrasive probes such as diamond-coated probes.

V. CONCLUSION

The patented circular mode[11] generates continuous and constant circular probe displacements at potentially very high velocities that are three orders of magnitude higher than conventional SPM velocities and that approximate to those met in realistic cases. Because the relative probe displacement is circular, the probe velocity is constant without acceleration, deceleration or discontinuation. Therefore, it offers new opportunities and new approaches of measuring probe-sample interaction especially when stationary states are required. The circular mode could be employed independently or could be combined with other SPM modes as for example conventional force-spectrum mode.

We have illustrated the interest of the circular mode by presenting its ability to (1) measure the influence of the sliding velocity on adhesion forces, (2) measure easily and instantaneously the friction-load dependence, and (3) generate fast consequent wear track.

Many other applications for characterizing surface properties could be visualized by the SPM community for their special needs by implementing the circular mode either on an AFM or on any other member of the SPM family (scanning tunnelling microscope (STM), scanning near-field optical microscope (SNOM), etc.). Therefore, this new SPM mode appears to be a powerful tool to get new insights in many research fields such as mechanics, physics, biology, or for metrological purposes.

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