A novel atomic force microscope operating in liquid with open probe unit and optimized laser tracking system

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Received June 4, 2009

A novel atomic force microscope (AFM) for large samples to be measured in liquid is developed. An innovative laser beam tracking system is proposed to eliminate the tracking and feedback errors. The open probe design of the AFM makes the operation in liquid convenient and easy. A standard 1200-lines/mm grating and a sheet of filter paper are imaged respectively in air and liquid to confirm its performance. The corrosion behavior of aluminum surface in 1-mol/L NaOH solution is further investigated by the AFM. Experimental results show that the system can realize wide range (20 × 20 (μm)) scanning for large samples both in air and liquid, while keeping nanometer order resolution in liquid by eliminating the tracking and feedback error.

OCIS codes: 110.0180, 180.0180, 180.5810.

doi: 10.3788/COL20090710.0891.

One outstanding feature of atomic force microscope (AFM) is its capability to measure surfaces in liquid environment. Hydrated samples can be observed directly in their native environments. Moreover, imaging in liquid can lower the adhesive effect on the sample and avoid problems caused by capillary forces[1,2]. This permits AFM a broad of applications in micro electromechanical system, material science, chemistry, and biology[3−7]. In fact, to image samples especially large samples in liquid is often desirable to AFM. Different types of AFMs have been proposed to meet various requirements[8−10]. The disadvantages of the conventional scanning-sample AFM operating in liquid include that the sample size is limited and operation in a liquid cell is really tricky, because extreme care must be taken to avoid spills on the scanner[11]. In contrast, the scanning-probe AFM is more suitable for liquid operation. For this AFM type, how to let laser beam track the cantilever precisely during its motion is a challenge. Tracking lens is then introduced to solve this problem, and it does work in keeping the light beams impinge on the cantilever[12]. However, the previous tracking systems would introduce distortions especially for wide range scanning.

In this letter, a novel AFM with open probe unit which allows large samples to be easily measured in liquid is proposed. An innovative tracking system is designed. With this system, the tracking and feedback errors can be eliminated effectively. The basic principle and setup of this AFM are introduced and experiments utilizing this equipment are present.

The schematic diagram of the AFM system operating in liquid is shown in Fig. 1. A laser diode (LD) and a position sensitive detector (PSD) are mounted on a stationary frame. The cantilever is installed at the focus of a small tracking lens which is attached to a tripod scanner. The beam emitted by LD is collimated and then focused on the back of the cantilever through the tracking lens and a transparent window. The reflected beam from the cantilever finally strikes the PSD through a focusing lens.

The transparent window is designed to absolutely immerse the AFM into liquid and make it scan under the liquid surface. As the surface tension of liquid is only exerted on the meniscus of the window, the effect of surface tension could be avoided on the AFM. Meanwhile, refracted beams only pass through the interfaces of liquid-solid and solid-air, not the interface of liquid-air, the serious effect of liquid surface vibration could be removed.

In previous scanning-probe AFMs, a narrow laser beam passed though a tracking lens and focused on the cantilever with different incident angles, and the motion of the lens would cause a tracking error (Fig. 2(a)). To solve the problem, we develop an optimized tracking method, as shown in Fig. 2(b). The effective aperture of the lens is 2.5 mm which is realized by adding an aperture stop before the lens (focal length: 3 mm). The diameter of the collimated beam is 3 mm, which is a little wider than the aperture of the tracking lens. After passing through the tracking lens, the excess beam is blocked by the aperture stop so that the imaging beam will keep the same incident angle and intensity on the cantilever even when the lens has a lateral displacement S during X-Y scanning. As a result, the tracking error can be eliminated accordingly.

The relative motion between cantilever and detector

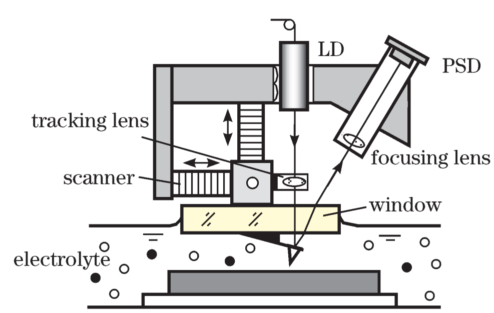

Fig. 1. Schematic diagram of proposed AFM probe unit operating in liquid.

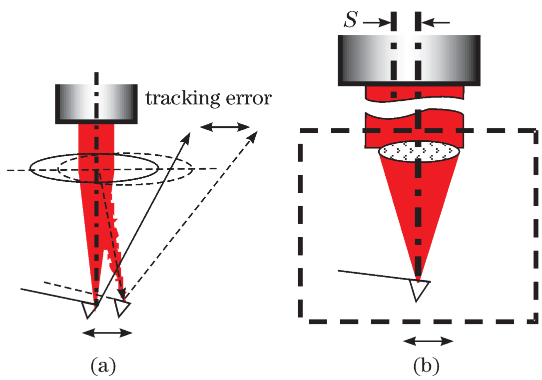

Fig. 2. Schematic of beam tracking system in scanning-probe AFM during scanning. (a) Previous method, (b) present method.

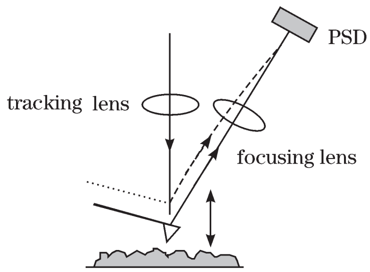

Fig. 3. Scheme of the optical path during feedback.

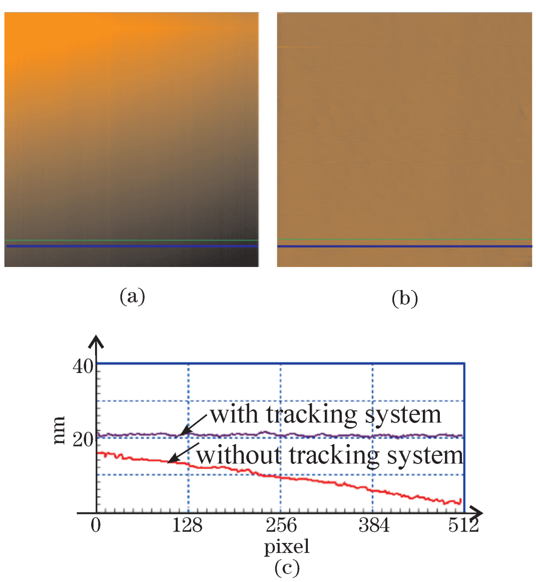

Fig. 4. AFM images (a) without tracking system and (b) with tracking system, (c) line profiles at the same position in (a) and (b).

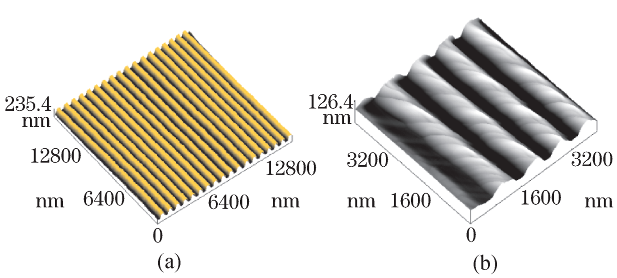

Fig. 5. AFM images of a standard 1200-lines/mm grating in air. (a) 16 × 16 (μm) scan, (b) 4 × 4 (μm) scan.

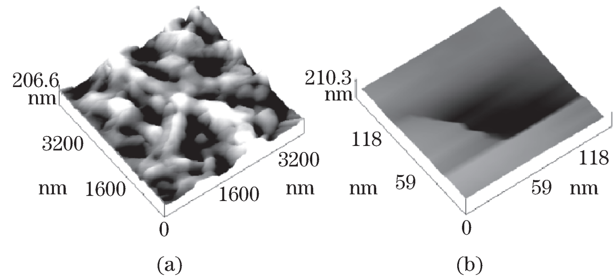

Fig. 6. AFM images of a sheet of filter paper in liquid. (a) 4 × 4 (μm) scan, (b) a hole in the surface (148×148 (nm)).

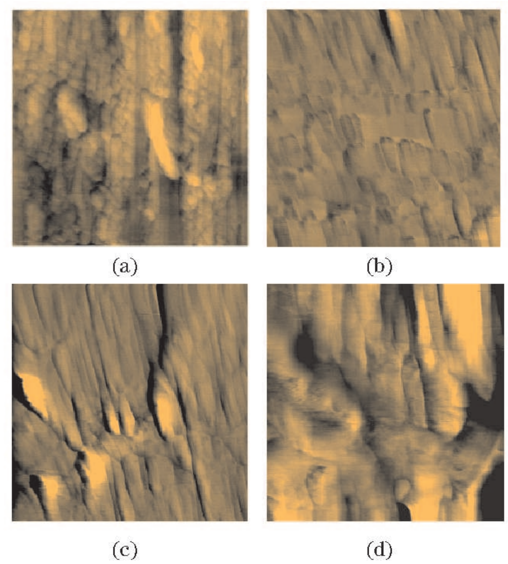

Fig. 7. Corrosion behavior of Al in NaOH solution investigated by the AFM (3 × 3 (μm) scan) at different time. (a) Beginning, (b) 15 s, (c) 90 s, (d) 150 s.

can also induce a feedback error. It makes the system get wrong response when scanning different places with the same height on the sample. As shown in Fig. 3, a PSD is fixed in the focal plane of the focusing lens (focal length: 3 mm). The translation motion of the reflected beams due to feedback will not change the spot position on PSD. With this method, the feedback error can be eliminated. Moreover, the cantilever is assembled on the object focus of the lens so that a large ratio of light lever can be guaranteed.

Some experiments were carried out to check the feasibility of the tracking system. Figure 4 shows the experimental result without (Fig. 4(a)) and with the tracking system (Fig. 4(b)). Figure 4(c) gives the line profiles at the same position in Figs. 4(a) and (b). It is found that the tracking and feedback errors can reach about 15 nm with previous method. In contrast, these errors can be reduced to less than 1 nm using our optimized tracking system.

Figure 5 shows the AFM images of a standard 1200-lines/mm grating scanned in air. The scanning range of Fig. 5(a) is 16 × 16 (μm). In order to view the detailed structure of the grating, a small range of 4 × 4 (μm) image is scanned, as shown in Fig. 5(b). Both of the images are of reliable stability, high resolution, and ideal contrast. It indicates that the system has satisfactory performance from wide range to small range.

To further demonstrate the performance of the AFM operating in liquid, a sheet of filter paper is imaged in distilled water, as shown in Fig. 6. A hole in the filter paper surface can be seen clearly in Fig. 6(b). It is demonstrated that the AFM measuring in liquid also has excellent performance, such as the nanometer resolution and repeatability.

As an example of application, the corrosion behavior of aluminum (Al) in 1-mol/L NaOH solution was investigated by our AFM system. To observe the detail clearly, a 3×3 (μm) region of the Al surface is on-spot scanned. Figure 7(a) presents the original image of the Al surface. After being put in NaOH solution, the Al surface exhibits pitting corrosion at first (Fig. 7(b)). Then crevice corrosion starts on the Al surface which leads to the width of the crevice increasing (Fig. 7(c)). After 150 s, the surface has been seriously corroded (Fig. 7(d)).

In conclusion, a novel scanning-probe AFM for large samples operating in liquid is developed. It is suitable for applications in aqueous or electrochemical environments. With a specially designed probe and an innovative tracking system, the instrument enables the cantilever to be totally immersed in liquid and the tracking and feedback errors can be eliminated effectively. The size and weight of samples are not restricted, which enables the instrument to image large or heavy samples. Applying this AFM, a standard 1200-lines/mm grating and a sheet of filter paper are imaged respectively in air and liquid. To further demonstrate the performances of the AFM, the corrosion behavior of Al is investigated in NaOH solution. Experimental results indicate that the AFM owns reliable stability, high resolution, and repeatability from wide range to small range, which can satisfy the requirement of measurements in liquid.

This work was supported in part by the National "863" Program of China (No. 2006AA04Z237) and the Open Subject of State Key Laboratory of Modern Optical Instruments (No. 200705).

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