Atomic Force Microscopy Utilizing SubAngstrom Cantilever Amplitudes

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(Received 8 November 2005; Accepted 26 November 2005; Published 20 January 2006)

A major problem of dynamic mode atomic force microscopy has been the necessity to employ relatively large amplitude of drive of the cantilever in the few Angstrom to the nm range to allow for sustaining self-excitation, and to obtain sufficient signal to noise ratio for frequency or amplitude shift measurement. The use of laser Doppler interferometry and super heterodyne signal processing has enabled clear atomic resolution imaging using subAngstrom cantilever amplitudes in the MHz regime. Due to the small amplitude, and the fact that the tip apex was always within the field of force gradient, long life of the tip was obtained, and frequency shift could more readily be interpreted as that coming from the short range forces. [DOI: 10.1380/ejssnt.2006.110]

Keywords: atomic force microscopy; low amplitude; lateral force microscopy; atomic resolution; cantilever

I. INTRODUCTION

When true atomic resolution atomic force microscopy (AFM) [1, 2] was accomplished in 1995, the typical amplitude used for the cantilever oscillation was 30 nm. The relatively low spring constant of the cantilevers then employed, and the noise level of the detection system necessitated such working parameters to be chosen. Since then, one issue in the instrumentation of atomic force microscopy has been to find a more favorable set of parameters for the cantilever as well as to improve the detection and signal processing scheme. The general trend is to use stiffer cantilevers in the few 100 N/m to the kN/m regime with smaller oscillation of drive [3]. Improvement of detection and signal processing methods was necessary to compensate for the possible degradation of sensitivity that comes from the use of stiffer cantilevers and/or smaller amplitudes. Recently, amplitude of drive has been reduced to the few Angstrom range by an improved optical lever method with lower noise level [4], or stiff force sensing device in the kN/m range [5]. Our work lies on the same trend of stiff cantilevers operating at smaller amplitudes, but with one difference. The natural frequency of the cantilevers is elevated to the MHz region, which acts at once in improving sensitivity and facilitating detection.

II. INSTRUMENTAL

A laser Doppler interferometer was used to measure the velocity of an AFM cantilever oscillating at 1.6 MHz, its second flexural mode. The use of Doppler measurement enabled improved carrier to noise ratio as the frequency of measurement increased. This was because velocity of the oscillator increased with frequency for a given amplitude. As the result, reasonable carrier to noise ratio was obtained to enable self-excitation and frequency shift measurement with sub angstrom amplitudes. The use of super-heterodyne signal processing also contributed to imaging with small amplitudes. The output of the laser Doppler interferometer was shifted to an intermediate frequency of 10.7 MHz and then subjected to frequency shift measurement, amplitude measurement and phase shifting. Figure 1 shows the schematic of the microscope [6]. Figure 2 shows the amplitude equivalent noise versus frequency [7]. The noise level decreases with increasing frequency. Figure 3 shows equifrequency shift images of Si(111)-7 × 7, taken with different amplitudes [8]. The set point of frequency shift was chosen to give better contrast. The results show that amplitudes less than 30 pm still gives atomically resolved images. The technique allows imaging with amplitudes less than an angstrom, where the tip is always within the short range forces, and the tip is within tunneling distance from the surface throughout the cycle of its oscillation. Figure 5 depicts an AFM with an inverted optical microscope. The optical microscope is used to image the cantilever and the sample, as well as to guide the laser beams of heterodyne laser Doppler interferometer and photothermal excitation. Heterodyne detection was effective in avoiding crosstalk between excitation and vibration measurement.

III. IMAGING OF VARIOUS DOMAINS ON QUENCHED SI(111) SURFACE, AND IMAGING IN LIQUIDS

Imaging in the second deflection mode with small amplitudes was easier than with the fundamental mode. We attribute this mainly to the low amplitude, though it is

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true that Doppler detection has a higher noise level at lower frequencies, such as at the fundamental deflection mode of 300 kHz, and that we have not exhausted all the possible imaging parameters. In many cases, a tip that did not yield atomic resolution images at the fundamental mode gave atomic contrast at the second deflection mode. Figure 4 shows some examples of imaging. Detailed discussion will be made in another publication [9].

As for imaging in liquids, a commercially available cantilever was attached on the bottom of a Petri dish with its
Amplitude dependence of images

| Frequency shift | Amplitude |
|-----------------|-----------|
| -60 Hz          | 0.12 nm   |
| -61 Hz          | 0.11 nm   |
| -84 Hz          | 0.088 nm  |

FIG. 3: Imaging of Si(111) with various amplitude of drive. A commercial AFM cantilever was oscillated at its second mode of deflection at 1.6 MHz.

FIG. 4: Various domains on Si(111) quenched surface. (a)Si(111)-c(2 × 8), (b) Si(111)-c(2 × 8)/√3 × √3, (c), (d) Si(111)-(1 × 1) imaged consecutively.

IV. CANTILEVERS

Imaging have so far been performed with the second deflection mode of a commercially available AFM cantilever. This was because smaller cantilevers were not yet available at the time of imaging. In dynamic mode imaging, it is preferable to use a tailored smaller cantilever with a higher natural frequency, for such reasons as: (i) wider choice of spring constant and natural frequency, (ii) to enable the use of the fundamental mode at elevated frequencies, and (iii) higher force and mass sensitivity. Conventional cantilever fabrication techniques, based on removal of the buried oxide layer (BOX) of a silicon on insulator (SOI) is not suited for cantilevers shorter than around 10 µm. Such problems are: (i) overhang of the cantilever support that arises from the etching of the BOX layer, (ii) connection of adjacent cantilevers by the SOI layer that is no longer supported by the BOX layer, resulting in coupling of modes of cantilevers in an array, and (iii) broad cantilever support resulting in contact of the cantilever support shoulders to the sample before the tip.

To avoid the problems, we have fabricated small cantilevers measuring less than 5 µm by micromachining cantilevers and their base separately and bonding them together afterwards. Silicon to silicon bonding was performed by heating the structure at 1000°C for an hour after the two parts were fixed by meniscus force of water. Figure 6 shows an example. The alignment process however was not easy, and the yield rate of the cantilevers was low. To avoid the problems related to alignment, dust, and bonding, a bellows structure was patterned together with the cantilever on the SOI. When the structure was left to dry after etching, meniscus forces acted to close the...
FIG. 5: An in-liquid AFM with an inverted optical microscope. A microscope objective is used to guide the two laser beams two the cantilever attached on the bottom of a Petri dish. Clear monatomic steps of mica was imaged in water.

FIG. 6: Cantilevers fabricated by silicon to silicon bonding of the cantilevers to their base. (a) Apex of the base before the cantilever is bonded. (b) chip array, (c) after bonding of the cantilever to the base. Bonding is performed once between the cantilever base array and the cantilever array. (d) as seen from the cantilever side.

V. SUMMARY

Heterodyne laser Doppler interferometry has proved to be suitable for high frequency low amplitude imaging with the AFM. The method is well suited for a cantilever whose size is close to the diffraction limit, and opens new possibility of high sensitivity mass detection of what is manipulated by the probe.

Acknowledgments

This study is supported in part by the Japan Science and Technology Agency. The authors thank the workshop of IIS, University of Tokyo for the fabrication of the AFM heads.
FIG. 7: A bellows structure that closes by meniscus force upon drying. The cantilever travels forward and fixes itself on the silicon substrate by silicon-silicon bonding.

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