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Scanning probe microscopy with quartz crystal cantilever

Takahito Ono,[a] Yu-Ching Lin, and Masayoshi Esashi
Graduate School of Engineering, Tohoku University, Aza Aoba 6-6-01, Aramaki, Aobaku, Sendai, 980-8579, Japan

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This paper reports a quartz crystal cantilever for piezoelectric vibration sensing in scanning probe microscopy (SPM). SPM imaging by a frequency modulation detection method is demonstrated in ambient atmosphere using a 22.5-μm-thick cantilevered AT-cut quartz crystal with metal electrodes on both sides. The spring constant of the cantilever is calculated to be 10 N/m. Despite the low electromechanical coupling between the piezoelectric effect and the flexural vibration, a high sensitivity of 0.07 nm/(Hz)$^{0.5}$ to vibration is achieved in the second flexural mode. The cantilever self-oscillates on the basis of piezoelectric detection, and offers short-term stability of within approximately 0.5 Hz in ambient atmosphere at room temperature. The force curve of the self-oscillating cantilever shows that the self-oscillation can be sustained even when in contact with a sample. © *2005 American Institute of Physics*. [DOI: 10.1063/1.2031937]

Scanning probe microscopy (SPM) techniques are essential tools in nanoscience, with application in an increasing range of fields such as nanomaterials, bio-science and nano-engineering. The environments in which dynamic SPM is required, such as ultrahigh vacuum, atmospheres, liquids, low temperatures, and high magnetic field, are also broadening.[1] In this respect, the operating environment may restrict the performance of the sensing element as the key component of the force sensor in SPM. For example, in optical detection at cryogenic temperatures, the scattered laser light from the sensor heats up the specimen. In such cases, a self-sensing function and negligible heat dissipation are required instead of a laser displacement sensor.[2] Furthermore, particularly in viscous fluids, a high quality factor (Q factor) is needed for high-sensitivity dynamic operation.[3] In such limited measurement setups, actuation and sensing by the probe itself is advantageous in terms of both simplicity of the instrument and sensing ability in difficult environments. Recently, a tuning-fork quartz crystal resonator, with both self-actuation and self-sensing based on the piezoelectric effect, has been employed for SPM.[4–9] This technique offers high stability in terms of resonant frequency and a large Q factor even in viscous fluids. However, the spring constant of the resonator is of the order of few thousand N/m,[7] which is much larger than that of a conventional silicon probe and results in reduced force sensitivity. Thus, a quartz crystal sensor with much smaller spring constant may offer higher sensitivity.

In this letter, SPM using a cantilevered quartz crystal sensor is reported. Despite the weak electromechanical coupling with flexural vibration in AT-cut quartz, the cantilevered sensor exhibits high sensitivity to vibration. The relationship between the vibration mode and the sensitivity is investigated, and topographic imaging using the proposed configuration is demonstrated.

The details of the fabrication can be found elsewhere,[10] and will be reported at length in the near future. Briefly, the starting material is a 100-μm-thick quartz crystal plate. After conventional photolithography, a 50-nm-thick Ti layer and 100-nm-thick Pt layer are deposited on the plate and patterned as electrodes. A Ni pattern as a mask is then selectively electroplated, and the quartz plate is subsequently etched by reactive ion etching (RIE). After metallization of the reverse side, quartz crystal cantilevers with a thickness of 15–25 μm are obtained. A sharp tip is achieved by attaching a sharp-tipped silicon beam to the end of the cantilever with conductive glue. Figure 1 shows a typical scanning electron microscope (SEM) image of the fabricated cantilever. The inset in Fig. 1 shows a magnified view of the mounted tip. The longitudinal direction of the cantilever corresponds to the *x*-axis (electric axis) of the quartz crystal.

Mechanical deformation of the quartz sensor induces surface charge due to the piezoelectric effect. This charge was measured using a current–voltage amplifier with a feedback resistance of 10 MΩ. The amplifier was connected to one electrode of the cantilever, and the other electrode of the cantilever was grounded. The amplitude of mechanical vibration was also measured using a laser Doppler vibrometer for comparison with the vibration detected by the piezoelectric effect. Actuation of the cantilever using the inverse piezoelectric effect was also possible, although with much lower amplitude (several nm). Therefore, another piezoceramic actuator was placed in the vicinity of the cantilever to induce cantilever vibration. The frequency response spectrum was obtained from the vibration signal using a lock-in amplifier

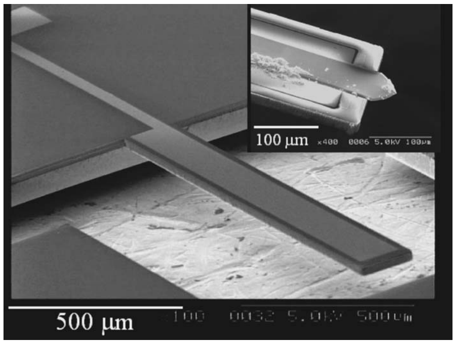

FIG. 1. SEM image of typical quartz crystal cantilever. Inset shows the sharp-tipped silicon cantilever mounted on the end of the quartz cantilever.

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[a] Author to whom correspondence should be addressed; electronic mail: tono@cc.mech.tohoku.ac.jp

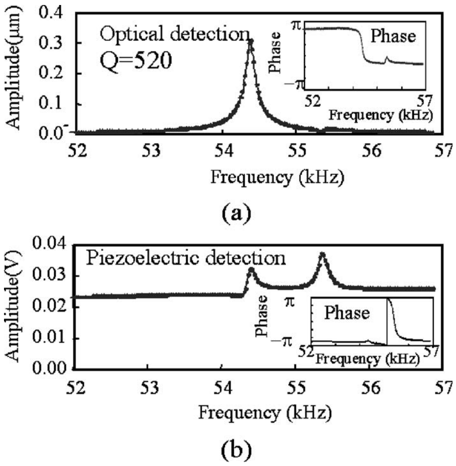

FIG. 2. Mechanical response spectrum of the 2nd flexural mode (a) measured by laser Doppler vibrometer and (b) measured by piezoelectric detection.

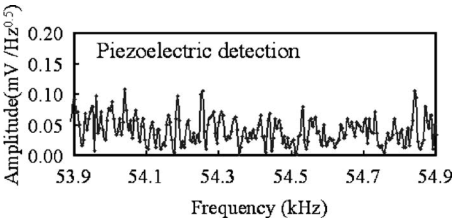

FIG. 3. Noise spectrum density of piezoelectric detection without vibration near the 2nd flexural mode.

and sweeping the actuation frequency. The noise spectrum density was measured using a spectrum analyzer. The vibration frequency was detected using a phase lock loop (PLL) circuit. Topographic imaging was performed using a laboratory-made SPM system. All measurements were performed in ambient atmosphere at room temperature (~300 K). The cantilever examined in this study was 1460 μm in length, 152 μm in width, and 22.5 μm in thickness.

The sensitivity of piezoelectric detection by the quartz cantilever was investigated by comparison of the laser Doppler vibrometer spectrum with the vibration determined from the piezoelectric signal. Using the laser Doppler vibrometer, the 1st, 2nd, and 3rd flexural modes could be identified at frequencies of 8.7, 54.2, and 142.1 kHz, along with fundamental torsional modes at 206.0 and 226.3 kHz. However, only the 2nd and 3rd flexural modes and torsional modes could be recognized in the piezoelectric spectrum. The sensitivity of piezoelectric detection exhibited a strong frequency dependence, with reduced sensitivity at low vibration frequencies, possibly due to stray capacitance. In AT-cut quartz crystal, only shear strain in the cantilever is electromechanically coupled with piezoelectricity.[11] The sensitivity of piezoelectric detection to vibration of the torsional mode is therefore larger than that for the flexural vibration mode. Despite the weak electromechanical coupling, however, a weak piezoelectric vibration signal could be observed in the 2nd flexural mode, as shown in Fig. 2. The Q factor of this mode is 520, and the signal is accompanied by a side peak at 55.3 kHz attributable to very small antisymmetric vibration in the longitudinal direction of the cantilever. The linearity of piezoelectric detection with vibration amplitude was found to be quite good from the measurement of various vibration amplitudes. At the peak resonant frequency of 54.2 kHz, the sensitivity is calculated to be 1 mV/nm. Figure 3 shows the noise spectrum density near the 2nd flexural mode. The mean noise density is approximately 0.07 mV/(Hz)$^{0.5}$, which is equivalent to a noise density of 0.07 mV/(Hz)$^{0.5}$. In a similar way, the measured sensitivity of piezoelectric detection for the 3rd flexural mode is 0.025 nm/Hz.

SPM imaging using the quartz cantilever was demonstrating using a frequency modulation (FM) detection method.[1] In this experiment, the piezoelectric detection signal from the cantilever was fed to the ceramic actuator via an amplifier, phase-gain adjuster, and bandpass filter to induce self-oscillation of the cantilever with a vibration amplitude of 30 nm. The frequency of the vibration signal was detected by the FM demodulator using the PLL. The error signal of the self-oscillation frequency from a set point was fed to the piezo scanner through a high-voltage amplifier. By adjusting the cut-off frequency of the bandpass filter for the self-oscillation loop, the cantilever tuned to self-oscillate near its 2nd flexural mode (55.3 kHz). As shown in Fig. 4, the short-term frequency noise of the self-oscillation was approximately 0.5 Hz with an integration time of 20 μs. The mode-dependent spring constants[12] of this cantilever for the 1st and 2nd flexural modes are calculated to be 10 and 1210 N/m, respectively. If thermomechanical noise is assumed to be dominant, the frequency noise Δf is given by $\langle(\Delta f)^2\rangle = f_n k_B T B / 2\pi k_n Q \langle z_{\mathrm{osc}}^2\rangle$,[13] where f_n and k_n are the resonant frequency and spring constant of the nth mode, T is temperature, B is bandwidth, k_{B} is the Boltzmann constant, Q is the Q factor, and z_{osc} is the amplitude of oscillation. Under the present experimental conditions, B was 140 Hz, Q was 520, and z_{osc} was 30 nm. With these parameters, the frequency noise due to the thermomechanical noise is calculated to be 0.003 Hz. As the measured value is 167 times larger than this calculated value, other noise sources in the detection circuit are considered to be dominant. The minimum detectable force gradient is given as a function of frequency noise as $\delta F_{\mathrm{min}} = 2k_n \Delta f / f_n$. According to this equation, the observed gradient for the 2nd mode is 0.02 N/m at a vibration amplitude of 30 nm, corresponding to a minimum detectable force of 6.0×10^{-10} N. The theoretical minimum detectable force F_{min} due to thermomechanical noise is given by $F_{\mathrm{min}} = (k_n k_B \mathrm{TB} / \pi f_n Q)^{0.5}$,[12] which yields a value of 3×10^{-12} N/(Hz)$^{0.5}$. If it becomes possible to use the first flexural mode of the cantilever, the minimum detectable force will be 4.3 times smaller than this value for the 2nd mode given the same Q factor.

A typical force curve for the self-oscillating in 2nd flexural mode operation is shown in Fig. 5. When the cantilever approaches the surface of a silicon substrate, a meniscus force due to a thin water layer on the surface attracts the

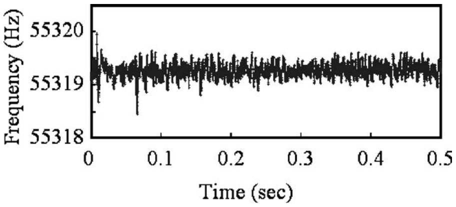

FIG. 4. Frequency noise of the FM demodulated signal of the self-oscillated cantilever with piezoelectric detection.

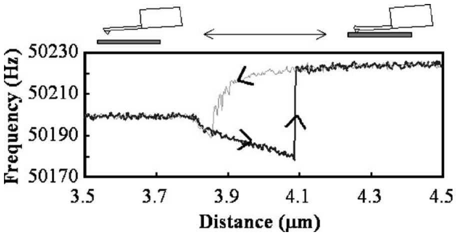

FIG. 5. Force curve of self-oscillating cantilever with FM detection.

cantilever, lowering the resonant frequency. Closer approach leads to physical contact between the cantilever tip and the surface, which causes an increase in the resonant frequency due to repulsive force. The meniscus force is accompanied by some hysteretic behavior. It is interesting that the self-oscillation can be sustained even when the cantilever comes into contact with the surface. This result suggests that imaging through a thin water layer will be possible using this cantilever.

SPM imaging of an oxide pattern on a silicon substrate was performed in the 2nd flexural mode at 54.5 kHz using this setup. The resultant image is shown in Fig. 6, where the step height is approximately 100 nm. These results demonstrate self-oscillation in the 2nd and 3rd flexural modes and the tortional mode, and imaging in these modes. The AT-cut quartz crystal vibrates on its x-axis in a shear vibration mode at a resonant frequency that increases with decreasing cantilever thickness. It is expected that strong electromechanical coupling with this shear mode and the high Q factor of the cantilever will provide high sensitivity and high stability of resonance for SPM applications.

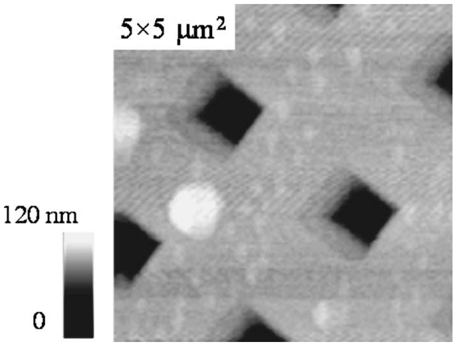

FIG. 6. SPM imaging of an oxide pattern on a silicon substrate measured using a FM detection method.

In summary, a cantilevered AT-cut quartz crystal with thickness of 22.5 μm for SPM was fabricated and characterized. The spring constant of this cantilever is only 10 N/m, and despite the low electromechanical coupling between the piezoelectric effect and flexural vibration, high sensitivity of 0.07 $\mathrm{nm/nm(Hz)^{0.5}}$ was achieved in the 2nd flexural vibration mode. The cantilever self-oscillates by the piezoelectric effect, and exhibits short-term stability of within approximately 0.5 Hz in ambient atmosphere at room temperature. The force curve for the self-oscillating cantilever shows that the self-oscillation can be sustained even when the probe comes into contact with the detection surface. SPM imaging was performed successfully using this cantilever in ambient atmosphere, demonstrating the potential of quartz crystal cantilevers with small spring constants for SPM and related technologies.

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