Surface Topographic Measurement Using Piezo–Electrical PVDF Film Stylus

Qiangxian Huang*, Non-member, Takeshi Hatsuzawa*, Member

A novel piezo-PVDF (Polyvinilidene Fluoride) film stylus has been developed to scan the surface topography for non-destructive and high-step measurement. The operating mechanism of the piezo-PVDF film stylus is similar to that of tapping mode SPMs. Instead of silicon cantilever often used in SPMs, a thin strip of piezo-PVDF film is adopted to form the vibrating cantilever here. Due to the flexibility of the PVDF film and the tapping mode, the scratching force is very small and non-destructive measurement is realized. By experimental measurements, a maximum error and a standard deviation along vertical axis are about 2.0nm and 0.5nm respectively. During an actual measurement on an epoxy grating using the measurement system with the single PVDF stylus, the repetability is proven to be within 4.3nm.

keywords: PVDF, tapping stylus, SPM, profilometry

1. Introduction

For the measurement of profile in the region of industry, high accuracy, non-destructiveness and high speed are pursued. After G. Binnig et al from IBM invented the AFM in 1986, the AFMs (Atomic Force Microscopes) have been improved to reach the resolution of better than sub angstrom and have very low measuring force of less than $0.1 \text{nN}^{(1)(2)}$. However, those AFMs usually cannot be applied directly to the samples surface with high aspect ratio in the region of industry due to the shortness of their tips. Although optical profilometers represented by optical microscopy profilometer have the advantage non-destructiveness and have the resolution of tens of nanometers, their horizontal resolutions are not better than sub micrometer due to the limitation of Airy Disk of objective lens. For contact stylus profilometers, measuring force of several millinewton should be applied to the styluses to ensure the tips to touch the surfaces of samples continuously. Therefore, the surface of the sample maybe scratched by the stylus easily and damaged.

Due to the disadvantages of AFMs, optical profilometers and contact stylus profilometer, our research group put forwards three methods that used quartz-fork, micro-fork and PVDF (polyvidine film) bimorph films as the tapping stylus respectively to measure the profile (3). The styluses touch the sample surface in the tapping mode just like the tapping mode SPMs (Scanning Probe Microscopes), therefore their measuring force is smaller than contact stylus profilometer; because their tips are about 1mm and far longer than that of AFM, samples with long aspect ratio surface can be scanned by those methods. Among the three methods, the quartz-fork stylus has the advantages of high vertical resolution (about 1nm) and low measuring force (about 1nN) but disadvantage of slow scanning speed; the micro-fork stylus has the disadvantage of rather larger measuring force (about 100nN); the PVDF bimorph stylus has advantages of high scanning speed and rather small measuring force (about 10nN), but it vertical

In order to keep the advantage of the above three styluses and overcome their demerits, a novel single PVDF film stylus is developed. Because this tapping stylus uses only one piece of PVDF film, its measuring force is only about 1/8 of that of bimorph stylus if the other parameters of PVDF were same ⁽⁵⁾. Because the structure is simple and stable, its vertical resolution is high. The following will present the operating principle, characteristic of the novel styluses, and also the measuring result using the new stylus.

2. Structure and Operating Principle of the Single Piezo-PVDF Film Stylus

Fig.1 shows the schematic structure of PVDF tapping stylus. A piece of PVDF film is bent and is fixed to vibration actuator on both ends. The vibration actuator is a piece of piezoelectric actuator driven by AC voltage. The fine tip is adhered by epoxy glue under the center of the PVDF, which is made of tungsten.

The vibration actuator, which is driven by ac voltage, vibrates along horizontal direction; Actuated by the vibration actuator, the

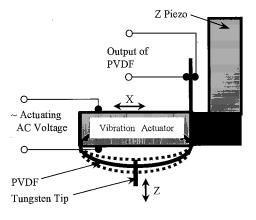


Fig. 1. Schematic and operating principle of PVDF tapping stylus.

resolution is low (about 10nm) because the stylus is unstable, which is caused by the bimorph structure (3)(4).

 ^{*} Tokyo Institute of Technology 4259 Nagatsuta-cho, Yokohama, 226-8503 Japan

PVDF cantilever and the tungsten tip vibrate along vertical direction. The frequency of ac voltage is changed until the PVDF is under the resonant vibration and the maximum of charge output is reached. In fact, in order to get the maximum sensitivity, the actual vibrating frequency is set below the resonant frequency, at which the actual output of PVDF is about 90% of its maximum. Then, the electric charge from PVDF film, which is piezo-electrical material, is amplified and sampled. While the stylus approaching the surface of sample, the vibrating state of PVDF is changed as soon as the stylus touches the surface. The output amplitude and phase of electric charge from PVDF decreases sharply. The decrement is decided by interference between stylus and surface of sample. If the sample is moved on an X-Y stage and the stylus is controlled up and down by the voltage applied on Z-piezo so that the decrement of magnitude or phase is on constant, then the voltage changing applied on Z-piezo is in accordance with the surface varying and the surface information can be constructed with the X-Y scanner. The operating principle is similar to other SPMs.

Compared with PVDF bimorph tapping stylus, single PVDF tapping stylus has advantages as following:

- 1) The touching force between stylus and sample is lower;
- 2) Since no adhesive is needed, vibration of PVDF is more steady and the noise more lower;
- 3) The structure is simpler;
- 4) Q is lower, higher speed maybe realized.

3. Determination of Parameters of the Single Piezo-PVDF Film Stylus

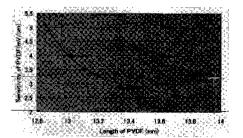
3.1 Design for the Parameters of the Stylus In Fig.1, piezo-electrical actuator AE0505D08 made by TOKIN Company is adopted. Its length, width and height are 10mm, 6mm and 6mm respectively. The PVDF film KF Piezo-Film made by KYNAR Company is adopted. The next step is to decide the optimal parameters value of PVDF and the optimal actuating voltage applied on piezo-actuator by experiment in order to obtain the highest sensitivity and lowest touching force. The parameters are the length, width and thickness of PVDF, the actuating voltage applied on piezo-actuator.

In tapping stylus, the distance between both ends of vibration actuator and its attachment is 12.8mm. The length of the vibration part of PVDF film is l, which is the part under the vibration actuator. If l is too long, the PVDF film is difficult to be vibrated, usually less than 13.5mm. If l is short, the PVDF film maybe touches the vibration actuator when it vibrates. Therefore, the optimum should be determined by experiment.

For the rectangle cantilever in SPMs, the spring constant can be estimated according the following formula:

$$k \approx M_e \rho whl \omega$$
(1)

where ω is the fundamental radial resonant frequency of the cantilever, w, h, and l are the width, thickness, length of cantilever, and M_e is the normalized effective mass, and ρ is the density of the cantilever. (5) Although this formula can not calculated the spring constant here, but it represents the relation between the spring constant and the dimensions of the stylus. Therefore, in order to reduce the measuring force, narrow PVDF film should be adopted. However, narrow PVDF film will decrease the sensitivity of the



(a) width: 2.1mm

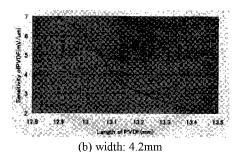


Fig. 2. Relation between sensitivity and dimensions of PVDF.

stylus.

In order to obtain the optimal parameter, we do a serial of experiments to observe the relation between the sensitivity of output and the length and width of PVDF film. Fig.2 shows the relation between vertical sensitivity and length of PVDF. Here, the thickness of PVDF is 40 µm, the peak-to-peak voltage applied on piezo-actuator is 12.5V, the width of PVDF is: 1.9mm in fig.2 (a) and 4.2mm in fig.2 (b). From the experimental data, shorter and wider PVDF film gives higher vertical sensitivity. But wider PVDF film causes larger touching force between the stylus and sample. Therefore, if the sensitivity of PVDF output is high enough, the narrower PVDF should be used.

For the rectangular cantilever spring constant, it can be explained by expression (1) and the following relations qualitatively:

$$M_e \propto I$$
(2)

$$I = \frac{wh^2}{12} \tag{3}$$

Therefore, in order to obtain small measuring force, thin film should be adopted. The PVDF film of thickness 40 μ m is adopted because the other one is 100 μ m.

The experiment also proves that higher voltage applied on piezo-actuator causes higher output, and the signal is easier to be picked up. However, if the voltage is too high, the quality of output wave from PVDF becomes worse, which maybe caused by noise vibration. From the experimental result, the applied voltage (peak to peak) should be less than 15V.

3.2 Frequency Response and Quality Factor Fig.3 shows the typical frequency response. The stylus is made based on the former obtained information. The output of PVDF is read by an oscilloscope directly, and the vibrating velocity of the stylus is measured through a Doppler velocity meter. The curve with triangular marks is the vibrating velocity of PVDF in free

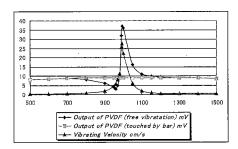


Fig. 3. Typical frequency response of the stylus at driving voltage 4V(peak to peak) and PVDF length, width, and thickness of 13mm, 2.1mm, and 40 micrometer.

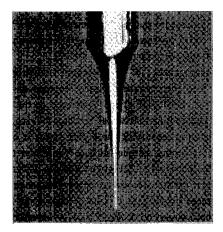


Fig. 4. Photo of a tungsten stylus probe Diameter of the tungsten wire: $60 \mu m$.

vibration and the curve with rectangular marks is the output of PVDF while the vibration of PVDF is limited by sample completely.

According to the experimental results, the basic resonant frequency is around 1kHz and the quality factor is about 40. In fact, after the tungsten tip had been adhered to the film, the basic resonant frequency is lowered to around 800Hz. The resonant frequency changes slightly in accorded with the length variation of PVDF film.

3.3 Preparation on Probe of the Stylus The probe is made of tungsten wire with diameter of $60\mu m$ through two electropolishing methods. One is an improved drop-off method put forward by K. Takahashi *et al* $^{(6)(7)}$. The diameter of the tip can reach 50nm. The other method is the improvement of the above one, and the diameter can reach 20nm. Fig. 4 is the photo of one of the prepared tips. It is obvious that this tip can be used to measure the surface with high aspect ratio.

Usually, the length of the tip is 1~1.5mm. It is adhered under the center of PVDF film (shown as Fig.1) by epoxy adhesive in room temperature.

4. Design for the Surface Measurement System with Single PVDF Film and its Parameters

4.1 Analogue Charge Signal Processing Circuit from PVDF Film The block diagram for analogue charge signal processing circuit from PVDF is shown in fig. 5.

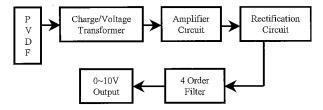


Fig. 5. Block diagram for analogue charge signal processing circuit.

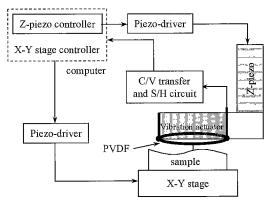


Fig. 6. Schematic of the surface measurement system with single PVDF film.

Actuated by the vibration actuator, the direct output signal from PVDF film is electrical charge. The signal is very small and can be attenuated by noise capacitance. In order to eliminate the noise capacitance from electrical element and wire, the electrical charge integral amplifier circuit is used to process the signal from PVDF film. Since the PVDF sensor is sensitive to temperature, wind and vibration *etc.*, the sensor and its lead should be sheltered from various types of noise. Before the charge signal is transformed into voltage, due to the small charge signal, the electrical elements should be chosen carefully, and the whole circuit should be covered by isolation case.

Because the frequency of carrier wave is very low, only about 800Hz, two filters of 2 orders are adopted after the signal is amplified and rectificated. One is a 2order Bessel low-pass filter with resonant frequency of 75Hz, the other one is a 2order Chebychev low-pass filter with resonant frequency of 136Hz. After being filtered, the carrier wave is less than 2mV, and a dc output between 0V and 10V can be obtain.

4.2 Block Diagram of Surface Measurement System

Fig.6 shows the schematic of surface measurement system. The system consists of computer, PVDF stylus including Z-piezo, vibration actuator and C/V transfer, and X-Y scanning stage driven by Piezo-driver.

The sample is fixed on a designed X-Y piezo-actuated scanning stage. The X, Y piezo-electrical actuators are same types used in the above-explained PVDF stylus. Its maximum driving voltage and maximum nominal stroke are 150V and $9\mu m$.

The operating principle of this system is as same as that of SPMs. The computer with the control software samples the signal from PVDF, and controls the movement of X-Y scanning stage. When the sample taps the surface of the sample, the output of the

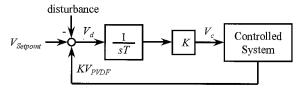


Fig. 7. Control model of the surface measurement system.

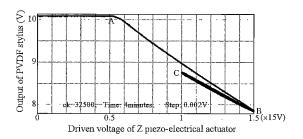
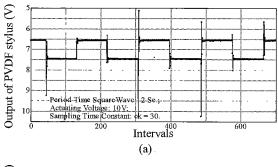


Fig. 8. Curve of output of the PVDF stylus and the voltage applied on Z piezo-electrical actuator.



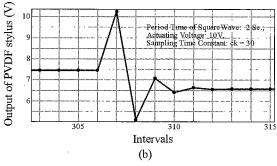


Fig. 9. Response of the PVDF stylus on step pulse (The step pulse to the stylus tip: about 700nm; Time Constant: about 5~6 intervals, 1 interval equivalent to 0.011ms).

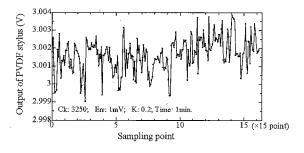


Fig. 10. Curve of noise in the output of the PVDF stylus.

PVDF stylus is the function of the gap between the tip and the surface. While the sample is moved along X-Y direction and the PVDF stylus is controlled to move up and down at the same time so as to maintain it's output at a setpoint, the surface information can be obtained through the driven voltage applied to the piezo-electrical actuator of the PVDF stylus.

4.3 Control Model and Software The movement of PVDF stylus in vertical direction is controlled by software and analogue circuit shown as Fig.5. It can be simplified as control model shown as Fig.7. Obviously, the height control system can be considered as a PI model.

In Fig.7, differential value V_d is calculated by software. Its discrete calculation is following:

$$V_{d(t+1)} = K(V_{PVDF(t+1)} - V_{PVDF(t)}) - V_{setpoint}$$
 (4)

Here, K is decided by the vertical sensitivity of PVDF stylus, the ratio of the Z piezo-electrical actuator's extension and driven voltage. In fact, the calculated value is about 0.2.

4.4 Determinations of the Vertical Sensitivity of the PVDF Stylus and the Dynamic Time Constant of the System The sensitivity of the PVDF stylus is defined as the ratio of the stylus output and Z piezo-electrical actuator's drive voltage. In order to estimate the sensitivity, the PVDF stylus is controlled to approach and leave the sample surface while the tip taps the surface, and the output of PVDF stylus and the driven voltage of the Z piezo-electrical actuator are recorded. The sensitivity can be estimated through experimental curves shown as Fig.8. The hysteresis of the Z piezo and drift of PVDF stylus are observed.

In Fig.8, the tip of PVDF stylus begins to tap the sample surface at point A. From point A to B, the tip is closing to the sample surface. From B to C, the tip leaves and is close to the sample surface for 3 times. The average vertical sensitivity of the PVDF stylus is the slope of straight line between A and B. Here, the sensitivity is about 0.12.

The sensitivity can be expressed as the ratio of the PVDF stylus' output and the decrease of the gap between tip and surface. In this form, the sensitivity of the PVDF stylus is about 1.98V/µm.

The time constant of the system is measured through step pulse shown as Fig.9. The theoretical highest sampling rate is estimated 15points/s.

4.5 Vertical Resolution of the PVDF Stylus and Temperature Drift Fig. 10 shows the noise in the output of the PVDF stylus when the stylus tip taps the sample surface at the same point. The sampling rate is about 4points/second. If the time drift is corrected, the maximum of the noise (peak-to-peak) is

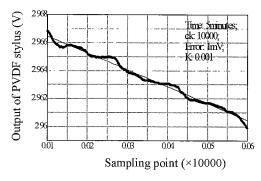


Fig. 11. Drift of the output of the PVDF stylus.

about 4mV. The standard deviation is about 1mV. Those two values in voltage are equivalent to about 2nm and 0.5nm, and considered to be the resolution.

Fig.11 shows the drift of the output of the PVDF stylus. By observation, the drift is caused by temperature and hysteresis of the piezo-actuator. The drift shown in Fig.11 is mainly caused by temperature because the piezo hysteresis mainly arises at the beginning (not be shown here). We have observed that the drift reached several hundreds millivoltage by 1°C changing of the air.

5. Experimental Results and Analysis

Using the PVDF film stylus, two types of samples is measured, one is epoxy grating, and the other is resist grating. The followings are the experimental results and repetability.

5.1 Experimental Result of Epoxy Grating and Analysis The epoxy grating is made of silicon with sputtering epoxy on the surface. It has a nominal pitch of $1.67\mu m$ and a nominal blade angle of $17^{\circ}27^{\circ}$ shown in Fig.12 (a), (b) is the SEM photo of a part of the sample and (c) is the 3D view with a scanning area of $8\mu m \times 5.8\mu m$. The sampling points is 127×20 points. The average sampling rate is about 4.5 points/s.

In Fig.12 (c), the average height of the peaks is about 0.52µm; the average pitch is about 1.68µm which is very closed to it nominal value; the angle between the right inclined plane and horizontal axis is about 15.9°; the angle between the left inclined plane and the horizontal axis is about 66°. The blade angle is smaller than its nominal value, this is due to the sample is inclined on the X-Y scanning stage, which it is obvious in Fig.12 (c). Although the inclined angle cannot be calculated, it is estimated to

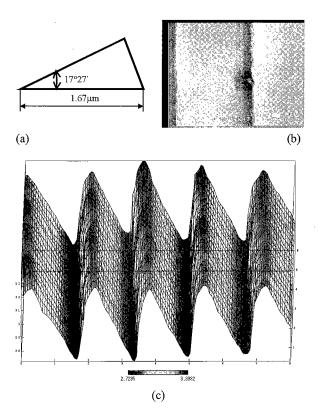
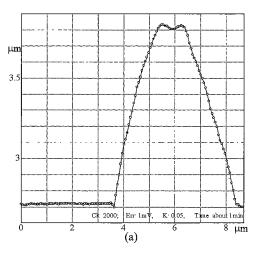


Fig. 12. Profile of epoxy grating. (a) Nominal dimensions of a pitch; (b) SEM photo; (c) 3D view scanned by the PVDF film stylus.



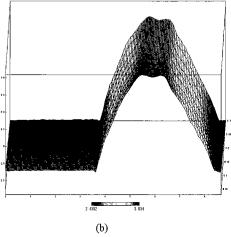


Fig. 13. Profile of resist grating. (a) A cross section; (b) 3D view scanned by the PVDF film stylus.

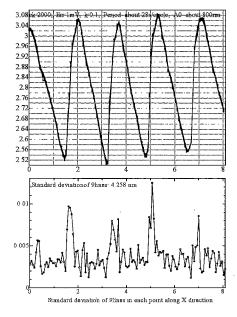


Fig. 14. 9 repeated curves of the same line (upper figure); Standard deviation of 9 times repeated measurement at each point (lower figure).

be between $1^{\circ}\sim 2^{\circ}$ based on the experimental data (This angle is also observed in Fig.14). According to the experimental result, the left slope proved that the PVDF stylus could scan the surface with large aspect ratio.

According to our observation, the sampling rate is about 4.0points/s.

5.2 Experimental Result of Resist Grating and Analysis The grating is prepared by us with the material OFPR-800 resist. The liquid resist material of OFPR-800 is coated on the silicon surface, and then the grating line is made through shining ultraviolet ray under special mask and being etched by chemical method.

The cross section and 3D view are shown in Fig.13 (a) and (b). The scanning area is $8.4 \mu m \times 2.8 \mu m$ with 137×10 points. Due to that the pitch exceeds the stroke of the X-Y scanning stage, only part of a pitch is observed. From the experimental data, the height in this scanned region is about $1.13 \mu m$. At the left bottom of the grating shown in Fig.13 (a), the maximum peak-to-peak value is 7.5nm including the roughness of the bottom and the measurement error.

In this measurement, the sampling rate is about 4.3 points/s, which is a little higher than that of in the above measurement. This is because the bottom of the resist grating is a plane and a shorter time is needed to let the stylus reach the setpoint.

5.3 Estimation of the Measurement Repeatability of the System

In order to obtain the repeatability, the epoxy grating is sample on the same line for 9 times. Fig.14 shows the 9 curves of the 9 times repeated measurement (upper figure); the temperature drifts between curves are corrected. The lower figure in Fig.14 is the standard deviation of 9 times repeated measurement at each point.

Through calculation, the average standard deviation of 9 curves, which corresponds to the repeatability, is about 4.3nm, far larger than the vertical sensitivity 0.5nm shown in Fig.10. It is considered that this is caused by the hysteresis of piezo-electrical actuators and drift of circuit. Comparing the shape of the upper curves and standard deviation curve (lower figure), the steep inclined planes (left) have larger standard deviation. This can be explained that the influence of the hysteresis and drift along X direction on the height of the PVDF stylus at steep slope is more serious than that of at gentle slope.

6. Conclusion

In order to detect the profile with high aspect ratio, a novel stylus using a single piezo-electrical PVDF film has been developed, and it is proved useful. Due to the flexibility of the PVDF film sensor and tapping mode, the damage on sample caused by tapping force is considered to be small. The PVDF stylus is very sensitive on vertical direction, and its maximum error and standard deviation of error are 2nm and 0.5nm.

Moreover, the designed system is used to detect two gratings, one with slope angle of 66° and the other with height of $1.13 \mu m$. The standard deviation of error in measurement of grating with slope angle of 66° and 15.9° is about 4.3nm after the temperature drift had been corrected. The time constant of the system is less than 70ms. The measurement accuracy is satisfied.

However, it is observed that the PVDF stylus is very sensitive to the temperature and environmental vibration. This should be treated carefully.

(Manuscript received July 10, 2002)

References

- G Binnig, C F Quate, and Ch, Gerber: "Atomic Force Microscopy", Phys. Rew. Let. 56, No. 9, pp. 930-933 (1986)
- (2) J D. Holbery, V. L. Eden, M. Sarikaya, and R M Fisher: "Experimental determination of scanning probe microscope cantilever spring constants utilizing a nanoindentation apparatus", Rev. Phy. Instrum., 71, No.10, pp.3769-3776 (2000)
- (3) T. Hatsuzawa and K. Takahashi: "A study on tapping styluses for surface profilometry", T. IEE, Japan, 120-E, No.3, pp.93-98 (2000)
- (4) T. Hatsuzawa, K. Ayabe, K. Takahashi, and M. Hayase: "A tapping stylus for surface topographical measurements using A PVDF bimorph", T. SICE, 35, No.9, 1141-1145 (1999)
- (5) John E Sader, James W. M. Chon, and P. Mulvaney: "Calibration of rectangular atomic force microscope cantilever", Rev. Sci. Instrum., 70, No.10, pp.3967-3969 (1999)
- (6) K. Takahashi, M. Hayase, and T. Hatsuzawa: "Improvement of production reproducibility of tungsten probe using electropolishing", T. IEE. Japan 120-E, No.4, pp.151-161 (2000)
- (7) A -D. Muller, F. Muler, M Hietschold, et al: "Characterization of electrochemicall etched tungsten tips for scanning tunneling microscopy", *Rev. Sci. Instrum.* 70, No. 10, pp.3979-3972 (1999)

Qiangxian Huang (Non-member) received a Dr.Eng. degree



from Hefei University of Technology, China 1998. He is currently working at Precision and Intelligence Laboratory, Tokyo Institute of Technology, developing surface measurement sensor.

Takeshi Hatsuzawa (Member) received his Dr.Eng. degree



from Tokyo Institute of Technology in 1995. After working at the National Research Laboratory of Metrology for 12 years, he is a Professeor of Precision and Intelligence Laboratory, Tokyo Institute of Technology.