Fabrication and characterisation of piezoelectric microcantilever probe

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This Letter focuses on the piezoelectric microcantilever probe fabrication processes. The sensing layer was lead zirconate titanate (PZT) thin films, prepared by sol–gel process, and formed on the $Pt/Ti/SiO_2/Si$ (100) substrate. The silicon-based microcantilever probe fabrication process is based on the microelectromechanical system technology. The compatible processes make the PZT thin films successfully fabricated on the silicon-based microcantilever. The size of piezoelectric cantilever is 450 μm in length, 70 μm in width and 12 μm in thickness. The spring constant of the cantilever probe is 41.28 N/m measured by the micro-force testing system. The first resonance frequency of the cantilever probe is 43.78 KHz. The low leakage current of the PZT thin films is 0.265 nA with the applied voltage of 1 V. The results reveal that the piezoelectric cantilever probe can be substituted for the traditional atomic force microscopy probe.

1. Introduction: The piezoelectric cantilever probe has a wide range of applications in nanotechnology, biosensors and scanning probe microscopy. Yegingil *et al.* [1] have used the probe to differentiate invasive malignant breast cancer from non-invasive ones. The most conventional atomic force microscope (AFM) used the laser beam deflection system, introduced by Meyer and Amer [2], where a laser is reflected from the back of the reflective AFM lever onto a position-sensitive detector. The multi-probe AFM system using lead zirconate titanate (PZT) cantilevers can markedly reduce the complexity of an ordinary AFM system [3].

The piezoelectric microcantilever probe can act as both sensor and actuator, which is a good alternative to conventional AFM. Lee *et al.* [4] fabricated a ZnO piezoelectric microcantilever with a high-aspect-ratio nanotip; yet the resolution was not enough for practical use because of the low piezoelectric constant of ZnO. Since PZT thin films have the highest piezoelectric constant, 20 times higher than ZnO and a lower leakage current among all piezoelectric materials available, it has become the best material for the piezoelectric microcantilever probe. Shibata *et al.* [5] demonstrated the capability of a piezoelectric sensor and actuator structure on a diamond AFM cantilever, which is hard enough and wear-resistant. Watanabe *et al.* [6] prepared the PZT thin films and applied it to AFM both as displacement sensors and as actuators. Lee *et al.* [7] fabricated silicon cantilevers integrated with heater and piezoelectric PZT sensors for a low-power scanning probe microscopy data-storage system. Kim *et al.* [8] fabricated the PZT cantilever array integrated with piezoresistor sensor for high-speed parallel operation of AFM, but the structure of PZT cantilever could not solve the problem of the coupling voltage, so it was proposed to be PZT actuator.

Our previous works have shown the fabrication of the tip on the Si (100) substrate [9] and presented the property of the cantilever with spring constant of 20–40 N/m [10, 11]. This Letter focuses on the design and fabrication of the piezoelectric microcantilever probe, which made the Si tip under dual protection in the follow-up processes. The fabrication processes and characterisation of the piezoelectric microcantilever probe were described in the following Sections.

2. Fabrication: The schematic diagram of the piezoelectric microcantilever probe is shown in Fig. 1. The PZT thin films were used as a sensing material, and it was prepared by sol–gel process [12].

2.1. Preparation of PZT thin films: The PZT thin films were prepared with the Zr/Ti ratio 53/47, which is at the morphotropic phase boundary. The PZT solution was spin-coated onto the $Pt/Ti/SiO_2/Si$ substrate. The spin speed was 3000 rpm. The obtained wet film was subsequently baked at 180°C and then heated at 350°C for pyrolysing the organic. Rapid thermal annealing was conducted at the temperature of 600°C. The thickness of the PZT thin films was about 1 μm.

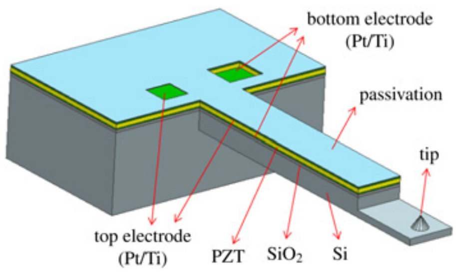

Figure 1 *Schematic diagram of the piezoelectric microcantilever probe*

2.2. Main fabrication processes of the piezoelectric microcantilever probe (shown in Fig. 2):

1. 1 μm-thick silicon dioxide (SiO_2) was formed by thermal growth on both sides of the silicon (100) wafer, which was 2 in. in diameter and 220 μm in thickness. SiO_2 oxidation is not only a mask for the silicon etching process, but also act as an insulator between the silicon substrate and bottom electrode.

2. The SiO_2 oxidation and exposed silicon on the back of the silicon wafer were etched by the wet etching process, while the top side of SiO_2 film were removed by HF solution.

3. The exposed silicon on the top of the wafer is etched anisotropically with SiO_2 as a mask. It was operated at 80°C for 20 min. A pyramid tip was formed on the top surface of the wafer.

4. Platinum (Pt)/titanium (Ti) bottom electrode was deposited by radio frequency magnetron sputtering technology. The thickness of Pt/Ti bottom electrode was 300 nm.

5. The PZT thin films were spin-coated.

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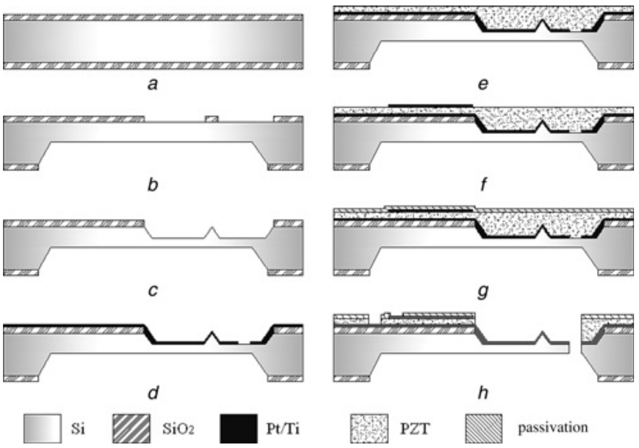


Figure 2 *Fabrication processes of microcantilever probe*
a Thermal oxidation
b Si and SiO_2 patterning
c Tip formation
d Bottom electrode patterning
e Deposited PZT thin film
f Top electrode patterning
g Preparation of passivation film
h Cantilever released

6. The Pt/Ti top electrode was deposited by the same method as used at the bottom electrode, patterned by the lift-off process. Fig. 3 shows the SEM image and cross-sectional illustration of finished piezoelectric thin films.

7. The PZT thin films were spin-coated for the passivation. The exposed silicon on the top was dry etched.

8. The cantilever was released.

The SEM image of the microcantilever probe is shown in Fig. 4*a*. The size of the probe is 450 μm in length, 70 μm in width and 12 μm in thickness. The length of the PZT sensing layer is 337.5 μm. Fig. 4*b* represents the SEM image of the silicon tip.

3. Results and discussion

3.1. Properties of the PZT thin films: The performance of the piezoelectric microcantilever probe strongly depends on PZT thin films. The obtained PZT thin films exhibit smooth surface and no crack. The crystallinity of the PZT thin films was characterised using X-ray diffraction (XRD) technique. The PZT thin films were well crystallised and have a perovskite structure as shown in Fig. 5. The deposited Pt electrode was optimised to <111> orientation, which led to the PZT thin films to have a preferred <111> orientation.

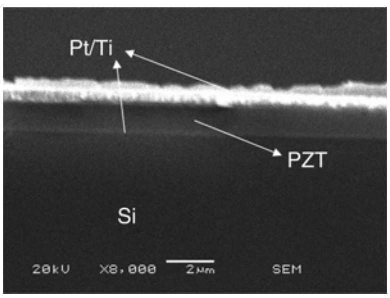


Figure 3 *SEM image of cross-sectional illustration of the piezoelectric thin films*

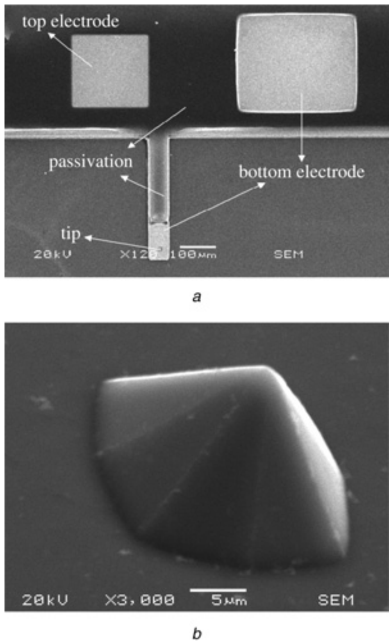


Figure 4 *SEM images*
a Piezoelectric microcantilever probe
b Silicon tip

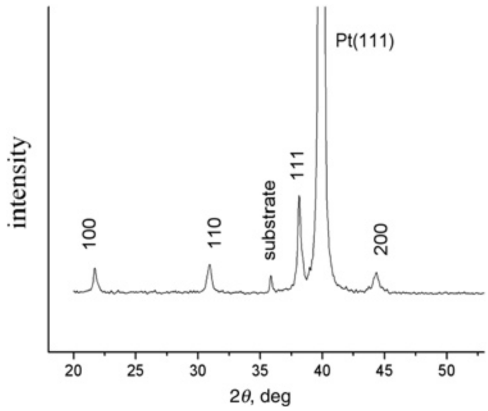


Figure 5 *XRD pattern of the PZT thin films*

The *P*–*E* hysteresis loop of the PZT thin films, which is shown in Fig. 6, was measured by Sawyer-Tower circuit. The PZT thin films were well saturated, and it was symmetrical with remanent polarisation value Pr of 8.39 $\mu C/cm^2$ and the coercive field value E_c of 67.47 kV/cm.

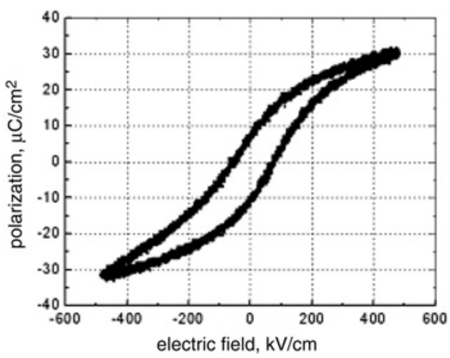


Figure 6 *P*–*E hysteresis loop of the PZT thin films*

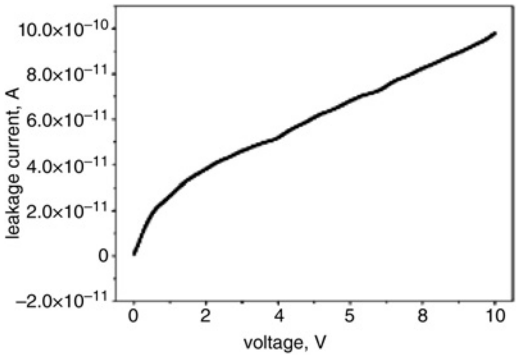
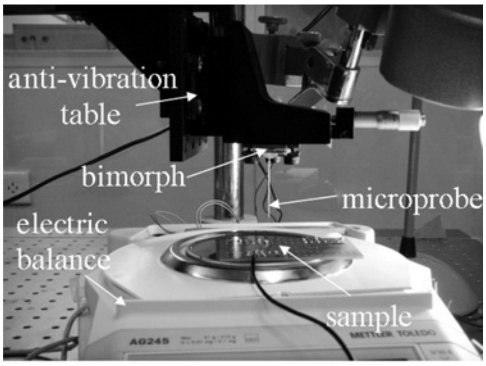


Figure 7 *I–V characteristic of the PZT thin films*

The leakage current of the PZT thin films was measured by semiconductor parameter testing instrument (Model 4200, Keithley). The leakage current was 0.265 nA, when 1 V DC voltage was applied to the PZT thin films. Fig. 7 shows the *I–V* characteristics of the PZT thin films.

3.2. Properties of the piezoelectric microcantilever: The resonance frequency of the piezoelectric microcantilever was measured by the AFM (CSPM AFM) in tapping mode. The first resonance frequency of the piezoelectric microcantilever is 43.78 KHz. The spring constant of the piezoelectric microcantilever was measured by the micro-force testing system [10]. Fig. 8*a* shows the micro-force testing system. The microprobe is fixed at the end of the bimorph, which contacts the front and back of the piezoelectric microcantilever. The DC voltage was applied and the bimorph is made to bend in order to generate micro-force. The electronic balance (AG245, Germany) measured the micro-force with 0.1 mg accuracy. The spring constant of the piezoelectric microcantilever probe is shown in Fig. 8*b*, which is 41.28 N/m.



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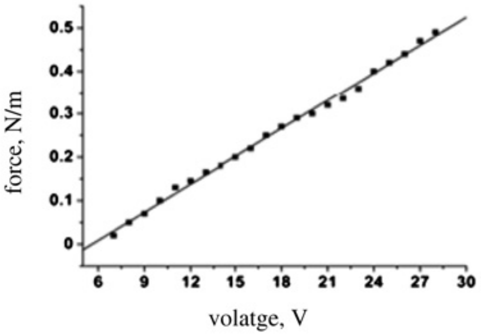

b

Figure 8 *Properties of piezoelectric microcantilever*
a Micro-force testing system
b Spring constant

4. Conclusions: The piezoelectric microcantilever probe was designed and fabricated. The fabrication processes mainly involve the silicon probe process, the PZT thin films process and the microcantilever process. The whole process has been optimised and compatible. Both the bottom electrode and the PZT thin films can make the Si tip under dual protection. So there is no damage to Si tip in the follow-up processes. The PZT thin film is fabricated by the sol–gel method. The property of the PZT thin films shows a good phase transformation to perovskite structure. The characterisation of the piezoelectric microcantilever probe shows that it is similar to traditional commercial silicon probe in the aspect of microcantilever sizes, resonant frequency and force constant. So, it can be a substitute for the commercial AFM probe in the property.

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