A Switchable Cantilver for a Chemically Sensitive Scanning Force Microscope

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We describe a cantilever device for a novel 'Time-Of-Flight Scanning Force Microscope (TOF-SFM)' concept that has the capability of chemical analysis. The cantilever device consists of a switchable cantilever (SC), a microfabricated extraction electrode, and a LEGO-type microstage, which combines two different systems. It allows quasi-simultaneous topographical and chemical imaging of a sample surface to be performed in the same way as with conventional scanning probe techniques. This is achieved by the micromachined SC with a bimorph actuator that provides a reasonable switching speed in comparison with mechanically operated switches. Secondly, a short tip-electrode distance to minimize the ions extraction voltage can be realized by LEGO-type microfabrication. The measured SC tip deflection is ~100 μm at 35 mW, corresponding to an estimated heater temperature of ~250℃. The maximum switching speed between the two modes is ~10 msec, and the sensitivity $\Delta R/R$ of an integrated piezoresistive deflection sensor is $\sim 6.7\times10^{-7}$/nm. The tip-electrode distance is only 10 μm. The TOF-SFM is currently integrated in an ultra-high-vacuum system to perform several measurements.

Key Words :

1. Introduction

Since the invention of the scanning tunneling microscope (STM) (Binnig et al., 1983) and the scanning force microscope (SFM) (Binnig et al., 1986), many STM- and SFM-based techniques have been developed for atomic imaging and manipulation (Eigler and Schweizer, 1990; Crommie et al., 1993; 1995, http://www.almaden.ibm.com/vis/stm/gallery.html). These new techniques have opened up entirely new possibilities for studying the structure and dynamics of individual molecules and atoms in surface science, biophysics, and the semiconductor industry (Wiesendanger, 1994; Tanaka and Kawai, 1996; Spong et al., 1989; Lee et al., 2001; 2002; Vettiger et al., 2000). In spite of striking achievements, these probe-based techniques have the disadvantage that they do not have the capability of chemical analysis of solid surfaces. This issue is being tackled by new instruments that combine STM/SFM with mass spectrometers (MS). A number of developments by universities and companies are in progress.

One technique exploits the atom probe (AP), first introduced by Muller in 1968 (Mueller et al., 1968), which was based on a field-ion microscope (FIM) and the MS. To increase the spatial resolution, the AP has been modified with various types of analyzers (Panitz et al., 1969; Mueller and Tsong, 1969; Chambers and Ehrlich, 1976; Nishikawa et al., 1981; Miller et al., 1996; Mueller and Sakurai, 1974; Panitz, 1978; Crezo et al., 1988; Miller, 1991; Blavette et al., 1993). Recently the AP was shown to be an ultimate MS

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for chemical analysis of solid surfaces (Miller and Smith, 1989). However, a serious drawback of this approach is related to the requirement that the sample needs to have the form of a very sharp tip. Often electron or ion milling techniques have been used to make such kinds of the sample tips. An additional the difficulty in the process is that sample materials are limited to semiconductors or conductors. Moreover the area being analyzed is limited to an extremely small area of the apex of the sharp tip. In order to solve these disadvantages, Nishikawa proposed a new AP called "scanning AP" (SAP) in 1994 (Nishikawa and Kimoto, 1994). The basic idea of the SAP is to use a microextraction electrode, which scans over a flat specimen surface with many microcusps. This aspect represents an attractive advantage of SAP compared with the conventional AP. However, the range of materials is still very limited as materials to be process with a dry etching technique because many microcusps are required on the sample surface.

At the same time, Spence proposed another approach called "scanning tunneling atom probe" (STAP) (Spence et al., 1996). He combined STM with a time-of-flight (TOF) AP. The basic configuration of the system is nearly same as those of an STM except that a sample stage can be removed by means of a wobble-stick to change from STM to TOF-AP mode in an ultra-high-vacuum (UHV) chamber. However, this system has two serious drawbacks to be solved. First, it requires a lot of time to mechanically move the sample stage to other places in the same chamber. Second, the tip cannot be repositioned to the same spot on the sample surface : this means that the system cannot acquire a topographical and a chemical image simultaneously.

In order to realize an ultimate microscope with MS, we propose a new chemically sensitive SFM called "Time-Of-Flight Scanning Force Microscope" (TOF-SFM). The TOF-SFM is based on a conventional SFM system for surface imaging with atomic resolution and a TOF-MS for chemical analysis of single ions. However, the TOF-SFM uses a new microfabricated cantilever device for switching between the two operating machines. A piezoresistive deflection sensor and a bimorph microactuator to switch the tip direction are integrated on the cantilever device. For the TOF-SFM, this approach has two important advantages. First, a local electrode with a tip-electrode distance comparable to the dimensions of the cantilevers significantly reduces the field-desorption voltage needed. Second, the switching time between SFM and TOF-MS modes is reduced by orders of magnitude using the integrated a bimorph microactuator. The TOF-SFM with the switchable cantilever device will potentially provide much better chemical and topographical information of solid surfaces.

2. Cantilever Device Design

The TOF-SFM basic concept and the cantilever device are illustrated in Figs. 1(a) and (b), respectively. It consists of a microfabricated single cantilever (SC), an extraction electrode (EE), and TOF-MS. The cantilever device is operated in an UHV chamber. A special holder with four springs for electrical contacts supports the cantilever device, and is placed on a piezotube scanner used for the SC tip approach. The holder enables an easy change of the cantilever device in UHV environments. The basic principle for the TOF-SFM is as follows ; While scanning the SC tip in contact with the sample, the tip grabs chemical compounds on the surface, is then switched in front of the EE, and, by applying a potential pulse between the tip and EE, the chemical compounds are ionized, extracted, and accelerated towards the TOF-MS for analysis. By integrating the cantilever actuation and placing the EE in close proximity to the tip, individual measurements can be done by orders of magnitude faster (millisecond range) than with current systems. Hence a chemical image of a sample surface can be obtained in reasonable number of milliseconds.

A schematic layout of the SC is shown in Fig. 2. The SC consists of a silicon cantilever with an in-plane tip, a piezoresistive strain sensor (PSS), and a bimorph actuator. It has length $l=410$ μm, width $w=80$ μm, and thickness $t=2.8$ μm. While

the tip scans over a sample surface, the integrated piezoresistive sensor, located at the fixed end of the cantilever to increase the deflection sensitivity, senses the deflection of the SC. The integrated deflection sensor eliminates having to align the laser to the cantilever end and to the position-sensitive photodetector (PSPD). This simplification facilitates operation in difficult environments such as an UHV chambers. The piezoresistive sensor used is based on boron-doped silicon, which exhibits a large piezoresistive effect. The design of the bimorph actuator is such that it maximizes its efficiency to limit the switching temperature. In addition, V-grooves were etched into the Si cantilever surface along the bimorph pad to soften the cantilever in this area and to improve the actuation efficiency because of the larger bimorph area of the V-groove structure. The bimorph bilayer consists of the Si cantilever and an Al layer evaporated on top of it. Heating is provided by the integrated resistor, which also consists of boron doped-silicon, underneath the Al layer. To prevent undesirable diffusion of chemical compounds at the tip when the SC is switched from the SFM to TOF-MS mode, a large gap is fabricated between the SC tip and the heater area.

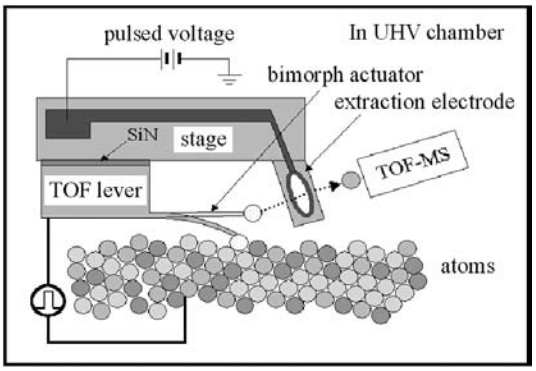

Fig. 1 (a) A concept of a chemically sensitive SFM and (b) a cantilever device used for tip switching purpose

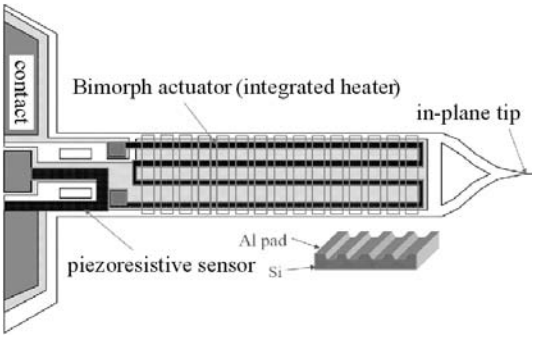

Fig. 2 Design of a switchable cantilever with an integrated deflection sensor

Figure 3(a) shows the process flow of key steps in the fabrication of the SC. A 4-inch silicon-on-insulator (SOI) wafer is used as starting material for the SC levers, the extraction electrodes

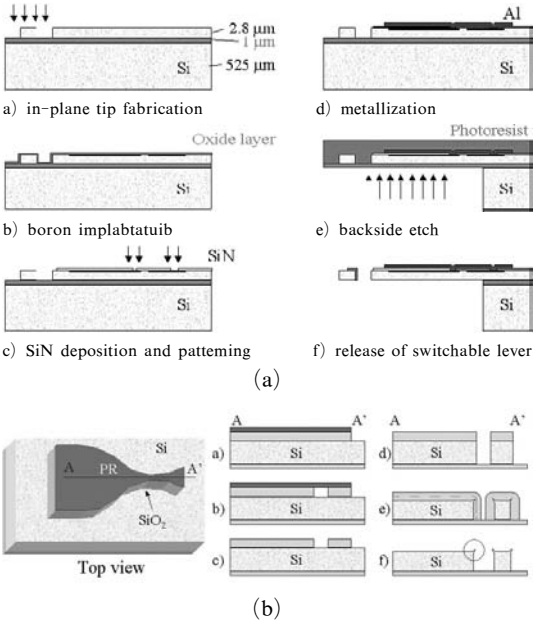

Fig. 3 (a) Process sequence of key steps in microfabrication and (b) the in-plane tip sharpening process by combination of two technologies, RIE and a low temperature oxidation

and the LEGO-type stages. All of them are made on the same wafer and with the same process. The initial substrate of the SOI wafer is an n-doped, 3-μm-thick silicon membrane. The cantilever with the in-plane tip is defined by photolithography and SF_6-based reactive ion etching (RIE, step (a)). Afterwards the tips are sharpened by a low-temperature oxidation technique. This technique is well known and has become an essential process for the tip sharpening (Akamine and Quate, 1992 ; Lee et al., 2002). The oxidation sharpening process is shown in Fig. 3(b). First, the shape of the in-plane tip is defined in SiO_2 by photolithography and CHF_3-based RIE (Fig. 3(b), step (a)). Then the SiO_2 is wet-etched until the neck of the base-shaped mask is 0 (step (b)). After removing the photoresist (step (c)) in acetone, silicon is dry-etched using SF_6-based RIE (step (d)). The silicon is thermally oxidized at 950℃ for 2 h (step (e)). The expected thickness of the oxide layer at the top of the surface is ~400 nm, which has experimentally been confirmed as being the best thickness for the tip sharpening. Finally, the SiO_2 is removed in a solution of buffered HF, which defines the in-plane tip at the free end of the SC (step (f)). A tip apex radius of 10 nm can be realized using this process. The shapes of the in-plane tip before and after the low-temperature oxidation are conformed with SEM images.

Two piezoresistors for the deflection sensor and the integrated heater are defined by boron-ion implantation at a density of 5×10^{15} atoms/cm^2 and subsequent thermal annealing at 1050℃ for 45 s (Fig. 3(a), step (b)). Then a stress-free SiN thin film is deposited by plasma CVD as electrical insulation between the Al layers and the piezoresistors. After defining the contact area by photolithography and the CHF_3-based RIE (step (c)), the Al pad for the bimorph structure and the metal wires for electrical connections are formed on the SC by a lift-off process using a negative photoresist (step (d)). SF_6-based deep RIE is used to pattern the back-side Si and to define the SC thickness (step (e)). Finally the SC levers are released by removing the topside protection photoresist in acetone (step (f)).

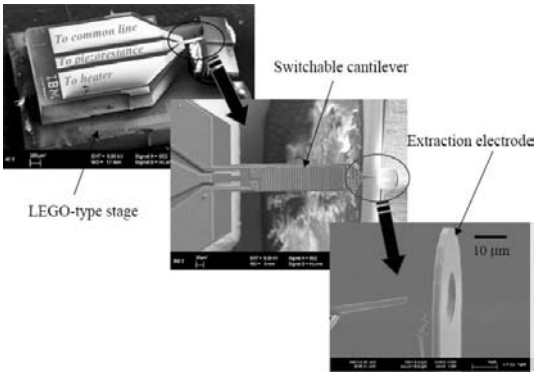

Fig. 4 (a) A fully assembled cantilever device, (b) a magnified image of the cantilever region, and (c) an image when the in-plan tip is switched to the extraction electrode

A SEM photograph of assembled SC/EE is shown in Fig. 4(a). The SC is pre-bent by controlling the deposition conditions of the different films on the SC (Al, silicon nitride). This provides the desired in-plane tip-approaching angle of 20° and a cantilever chip-sample clearance of ~100 μm while scanning. The microfabricated extraction electrode and the LEGO-type stage are batch-fabricated on the same wafer. The SC and the EE are then manually assembled and glued onto the microstage. The micromachined special holder with "LEGO-type" alignment structures is used to facilitate and improve the alignment precision of the assembly. Figures 4(b) and 4(c) show SEMs of the magnified tip/EE area and the tip position when it is actuated by the bimorph actuator. The distance between the tip and the EE is set to a few tens of microns to minimize the ion extraction voltage.

3. Experimental Results

In order to measure the resonance frequency of the SC, it is placed on a piezoelectric actuator, and two electrical contacts on the SC are connected to an external dc-biased Wheatstone bridge that directly determines the cantilever deflection by measuring the change in the cantilever resistance. The values of the piezoresistor and the three metal film resistors used in the Wheatstone bridge are ~650 Ω. A network analyzer measures

the frequency dependence of the SC. The measured resonance frequency of the SC is ~17 kHz, which is very close to the predicted value of ~20 kHz. The SC tip deflections as a function of actuation power for SCs with and without V-grooves have been measured. Note that the SC tip deflection is 1.5 to 2 times larger for the SC with V-grooves. The deflection improvement for the V-groove SC results from the optimized thickness ratio between silicon and aluminum, and the areal increase of the aluminum-pad V-grooves.

When the integrated heater is heated with more than 100 mW, Al melting is observed at ~110 mW. The Al melting point (660 ℃) is used as reference to calibrate the temperature vs. electrical power. A measured SC tip deflection of ~60 μm was achieved with 25 mW, which corresponds to a heater temperature of ~200℃. When the bimorph is heated with more than 40 mW, an additional remnant stress is observed in the SC, which is probably due to Al recrystallization and interaction with Si and oxygen, which occur at temperatures above 300℃. The frequency response of the cantilever deflection of the SC upon application of 25 mW actuation power is measured with an optical microscope and a function generator to decide the maximum switching speed of the bimorph actuator. The resulting switching speed of ~10 msec is very attractive for the TOF-SFM application. It is more than 100 times faster than currently available bulky actuators.

To obtain a surface image in non-contact mode, the SC with integrated PSS has been installed into the conventional SFM system. Two electrical wires from the PSS are connected to an external Wheatstone bridge circuit, which senses the cantilever deflection by measuring the change of resistance value. A sample for SFM calibration is imaged by the SC.

Figure 5 shows a topographic image of a standard sample used for SFM. The sensitivity of the integrated PSS is measured by bending the cantilever with a micromanipulator and measuring the change in resistance. The measured sensitivity $\Delta R/R$ is $\approx 6.7\times10^{-7}$/nm, which results in a gauge factor of ~26.

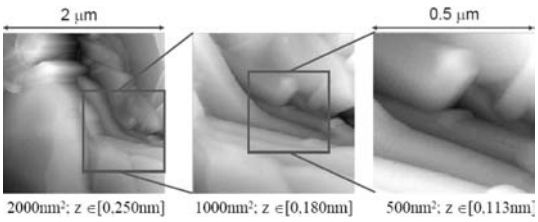

Fig. 5 Topography of a standard sample with SC

For the field emission experiments, the tip is switched to a position of a TOF mode by using the bimorph actuator and is grounded by a pico-amperemeter. A positive dc voltage on the EE is slowly increased to measure emission behaviors. The measured emission current that flows from the tip to the EE in vacuum is plotted as a function of applied voltage. Figure 6(a) shows the

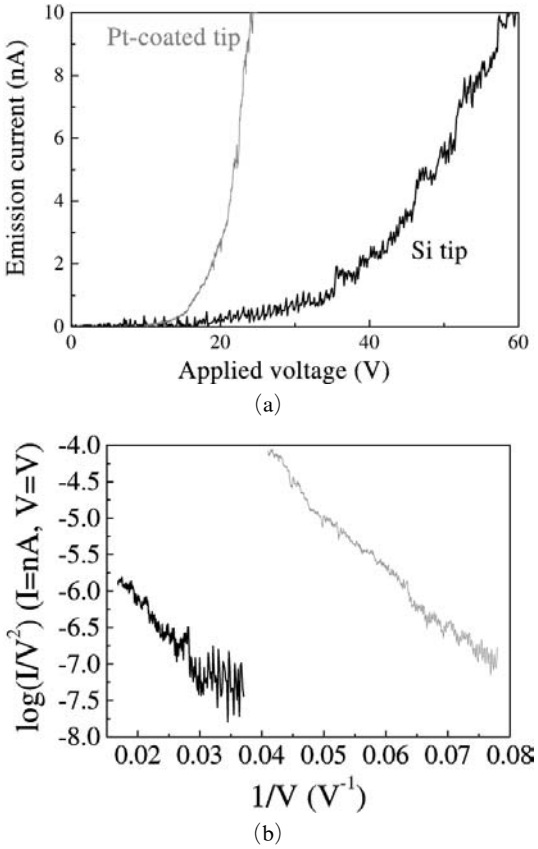

Fig. 6 (a) Field emission measure before and after a contact process onto a HoPG surface and (b) TOF-MS measurement with a W tip

field emission characteristics of two different conditions of tip, a Pt-coated Si tip and a pure Si tip. Figure 6(b) shows a Fowler-Nordheim plot of the $I-V$ curves. The Pt-coated tip turns on at only 15 V_{dc} thanks to the low Pt work function, the field enhancement due to the sub-20-nm tip radius, and the small tip-EE distance of less than 10 μm. The turn-on voltage of the SC with the integrated EE is less than 50 times that of the SC with a funnel-shaped Al electrode. The tip-EE distance for the funnel-shaped Al electrode was approximately 10 μm. This field emission experiment is of particular interest because tips start to field-evaporate at approximately the inverse tenfold electric field used for the field emission.

To measure the flight time of evaporated ions from the tip apex the SC with Pt-coated tip is placed into the TOF-SFM chamber. A negative pulse is applied to the EE using a Blumlein-type ns-kV pulser while a positive dc high voltage of 800 V is biased to the tip. The negative pulse amplitude is kept at 20% to 30% of the positive dc voltage value, which is constantly and slowly increased until the first ion impact is detected on a multichannel plate (MCP). The pulser defines the departure time t_0 of ions, and the MCP defines the arrival time t of the evaporated ions. Masses and chemical properties of the ions can be calculated using a computer system with labview control. The capability of single-ion detection in our TOF-SFM system is about 60%. The vacuum in the chamber during the TOF measurements was $\sim 3\times10^{-8}$ Pa. A TOF spectrum of the Pt-coated tip consisting of 30 pulses at $V_{dc}=800$ V and $V_{pulse}=-204$ is shown in Fig. 7. $C^+(12)$, $Si^+(28)$ and $Pt^{2+}(97.5)$ ions evaporated from the Pt-coated tip are observed in the mass spectrum when a negative pulse voltage is applied to the EE. Sufficient electric field for ion evaporation is achieved only at the moment of the negative pulse on the EE. We used a focused ion beam to deposit the Pt at the apex of the tip. Hence some of unknown ions are also observed on the figure, which is caused by the several impurities in a reaction chamber of the FIB system.

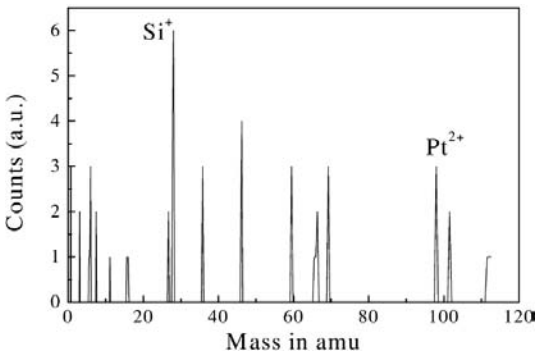

Fig. 7 TOF-MS measurement with the same tip after the contact process

4. Conclusions

We have fabricated and characterized a switchable cantilever for a scanning force microscope with chemical sensitivity called "TOF-SFM". The cantilever device is based on a single cantilever that combines the force sensor and the extraction electrode. The deflection efficiency of the SC is considerably improved by using a V-grooved bimorph actuator. SC deflections of ~ 60 μm have been achieved with an electrical power of 25 mW, corresponding to an estimated temperature of 200℃. The maximum switching speed of ~ 10 msec is demonstrated using the bimorph actuator, which is very attractive for the TOF-SFM applications. A short tip-electrode distance of less than 10 μm could be achieved by employing a microfabricated LEGO-type stage. The turn-on field emissions have been demonstrated with an extraction voltage as low as 15 V by using MEMS technology. The emission current and the TOF analysis have also been performed using two different types of cantilever devices with a Si tip and a Pt-coated Si tip (Lee et al., 2004).

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