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Silicon cantilevers and tips for scanning force microscopy*

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Abstract

Monocrystalline silicon cantilevers with integrated silicon tips for scanning force microscopy are fabricated by means of micromachining techniques Theoretical considerations including finite element modelling have been carried out in order to find a suitable shape and dimensions according to the mechanical requirements Several different cantilever designs have been fabricated a simple beam with various cross sections as well as a folded meander shape with square cross section Special attention has been paid to the application of these silicon microprobes to measure friction Moreover, high-aspect-ratio silicon tips with variable geometries are presented and their integration onto cantilevers is demonstrated Finally, the fabrication of an array of such microprobes is described, which enables multiple parallel or serial surface profiling to be achieved These integrated micromachined cantilevers have been successfully applied in standard atomic force microscope measurement systems

1. Introduction

Since the invention of the scanning tunnelling microscope (STM) in 1982 and its derivative the scanning force microscope (SFM), also known as the atomic force microscope (AFM), in 1986 [1], this class of microscope has proved to be an excellent tool for analysing surfaces with atomic resolution This opened the possibility of using an alteration of the atomic constellation as a storage device or as surface preparation for catalytic purposes The first steps of this engineering on an atomic level have already been undertaken [2, 3] In microelectronics itself such STM/SFMs are employed as profilers Thereby the chip surface is measured in three dimensions, which in a comparison with an ideal chip allows quality control and even the localization of defects to be carried out For this application it would be very important to have arrays, which is only possible with batch-fabricated heads

Force microscopy, whose operation principle has been widely published [e g , 4–8], is an extension of the STM, which makes it possible to investigate insulating materials as well a tip of a flexible force-sensing cantilever stylus is raster scanned over the surface of the sample under investigation The forces acting between the tip and sample cause minute deflections of the cantilever, which are detected and displayed as an image with very high spatial resolution Such applications are not confined to inorganic surface analysis, but extend even to biomedicine The polymerization of fibrin could be depicted *in situ*, e g , [9]

The requirements for accurate force measurements are low force constants (≈ 1 N/m) for high sensitivity and high resonance frequencies (> 10 kHz) for noise filtering These requirements were first achieved with skilfully prepared microprobes, like cut or etched metal wires and foils By silicon micromachining, thin-film cantilevers of silicon dioxide (SiO_2) with a glued diamond fragment as the tip [10] or silicon nitride (Si_3N_4) with an integrated tip [11] were microfabricated Polycrystalline silicon cantilevers using sacrificial layer techniques [12] and monocrystalline silicon (m-Si) cantilevers using bulk micromachining have also been presented [13, 14]

The tip–surface force interaction can be monitored quasistatically by measuring the cantilever

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deflection, e.g., via a tunnelling tip [1], an optical laser beam deflection [15, 16], capacitively [17] or with integrated piezoresistors [18]. A dynamic operation mode is also possible, and sometimes preferable, by driving the cantilever near its resonance frequency and measuring the frequency shift in the presence of a force gradient [3, 4].

In the field of force microscopy some interest has arisen in measuring not only the force in the z-direction but also the force parallel to the scanning direction (say the y-direction), especially in studying the tribological behaviour of surfaces (friction, wear and lubrication). This interaction can be measured either by monitoring the lever's torsion as a reaction to lateral forces on the tip or, when disposing of a symmetrically built cantilever, by measuring its sideways deflection. The types of cantilever construction for these two approaches are obviously not identical. For torsional measurements, a flat cantilever with low torsional rigidity (see below) is required, whereas sideways bending implies the stiffnesses in the z- and y-directions are similar.

Such an approach has been published by Neubauer *et al.* using a round wire with iridium, tungsten and diamond tips and utilizing a capacitive displacement sensor [19]. For a micromachined cantilever a square cross section would be ideal. This is no longer achievable with thin-film cantilevers, since the width of the beam would have to become smaller than 1 µm. This is not feasible with conventional photolithographic techniques and would complicate the readout even more.

Therefore we have fabricated m-Si cantilevers by using bulk micromachining techniques with different geometries, simple beams as well as folded ones, and include finite element modelling (FEM) studies carried out with ANSYS© [20] for the mechanical analysis and optimization of the structure. Then we show the fabrication of sharp silicon tips with different heights and aspect ratios and demonstrate their integration onto the silicon cantilevers. Finally, we demonstrate the suitability of batch fabrication for obtaining arrays of microprobes, enabling parallel surface profiling and an increase of the scan area versus scan time to be achieved or serial multiple-measurement to be carried out.

These integrated micromachined cantilevers have been successfully applied in standard AFM measurement systems.

2. Design of micromachined silicon cantilevers

2.1. *Simple cantilevers*

Micromachined cantilevers from Si_3N_4 or SiO_2 thin film were originally designed to have a high sensitivity in the vertical z-direction only and to be very stiff in the orthogonal directions y and x (inherent to thin film). By using a triangular cantilever this effect could even be increased. In general, the spring constant c_z of a simple rectangular beam cantilever is given by

$$c_z = Ewt^3/4l^3 \tag{1}$$

with E being Young's modulus of the cantilever material and w, l and t the lever's width, length and thickness, respectively. When aiming for a spring constant of typically ≈ 1 N/m and assuming typical measurement forces in the range 10^{-9} to 10^{-6} N, the resulting deflections of the cantilever are detectable by the readout systems described above. The bending is given by Hooke's law as $\Delta z = F/c_z$. The first flexural resonance frequency of such a structure can be expressed by

$$f_0 = 0.16\frac{t}{l^2}\left(\frac{E}{\rho}\right)^{1/2} \tag{2}$$

with ρ being the material density. For dynamic measurement of the frequency shift Δf as a function of a force gradient F' we find [10]

$$\Delta f = \frac{f_0}{2c_z}F' \tag{3}$$

We designed simple m-Si ($E = 1.69\text{E}11$ N/m^2, $\rho = 2300$ kg/m^3 [21]) beams in various sizes feasible for standard microfabrication techniques to have different mechanical characteristics, as summarized in Table 1.

2.2. *Lateral movement*

Lateral forces occurring during contact-mode measurements can be monitored by the cantilever's sideways bending Δy related to its lateral stiffness c_y by $\Delta y = F/c_y$, where

$$c_y = Etw^3/4l^3 \tag{4}$$

This is identical with c_y when using a quadratic cross section ($w = t$) (cf. eqn. (1)).

In order to find a compact shape with the required specifications and to increase the bending sensitivity, we compared analytically simple beams with cantilevers of various folded forms. With

TABLE 1 Mechanical characteristics of silicon cantilevers with different geometrical shapes and dimensions ($E = 1\ 69E11\ N/m^2$, $G = 0\ 5E11\ N/m^2$ $\rho = 2300\ kg/m^3$), for the calculation of c_t a tip length r of 20 µm is assumed

Length L[µm]	Width w[µm]	Thickness t[µm]	Spring constants c_z[N/m]	c_y[N/m]	c_x[N/m]	c_t[N/m]	1st mode f_0[kHz]
Simple beam							
700	15	15	6 24	6 24		0 060	42
700	30	10	3 70	33 26		0 036	28
500	30	10	10 14	91 26		0 050	56
Meander							
700	15	15	0 79	1 02	20 4		12

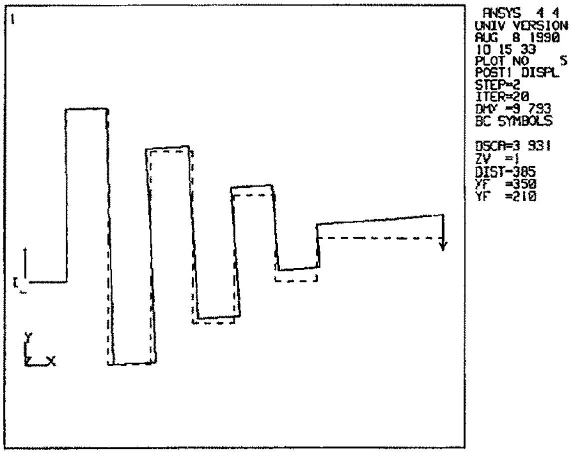

Fig 1 Flexural simulation of a silicon meander with dimensions $l = 700$ µm and square cross section of 15 µm × 15 µm, applied force 10 µN

Fig 2 SEM photograph of a silicon meander corresponding to that of Fig 1

ANSYS[1] we simulated a cantilever with a square cross section of 15 µm × 15 µm and 700 µm long, folded as a meander as illustrated in Fig 1 Thus the protruding length of the microprobe could be shortened up to half of the length compared to a simple beam with the same cross section and force constant The corresponding microfabricated meander is shown in Fig 2

Since the maximal stress in both the folded and straight bent beams occurs at its clamping and is determined by its overall length times the applied force at its end (bending moment), a weaker (folded) spring bends more than a stiffer (straight) one with the same cross section until fracture The folded cantilever can thus be made more sensitive for a given force range

For this shape it is also possible to adjust the ratio of the stiffness in the y- and z-directions to a certain degree (from 1 for a simple beam to 1 5 for a meander with a maximal width similar to the protruding length), since one movement is rather bending and the other torsionally determined The stiffness in the x-direction is, as required, more than one order of magnitude higher than in the measuring direction Note that the clamping of the structure is not at the central line, which decouples the y-movement from the x-movement, due to the asymmetric construction

2 3 *Torsional movement*

Another way to measure lateral forces is to monitor the cantilever torsion φ, which is related to the torsional rigidity c_t by $\varphi = M/c_t$, where

$$c_t = Gwt^3/3lr \tag{5}$$

G denotes the shear modulus (0 5E11 N/m^2 for silicon [21]) and r the tip height Depending on the geometry of the cantilever cross section as well as on r, the overall reaction is either more bending or torsionally determined When assuming a rigid tip

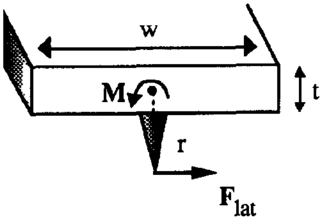

Fig 3 Illustration of the effect of lateral forces on the tip

(i e , not behaving like a spring itself) the lever's reaction will certainly be a superposition of a bending and a torsional movement, since the force vector does not go through the centre of rotation but is applied at the tip's end A torque of $\boldsymbol{M} = \boldsymbol{r} \times \boldsymbol{F}_{lat}$ thus acts on the lever (see Fig 3)

As can be seen from eqn (5), a flat cantilever ($w \gg t$) with a high tip results in a lower torsional rigidity than one with a square cross section ($w \approx t$), assuming the same surface area $w \times t$ By comparing eqns (4) and (5) we can evaluate the coupling of the lateral bending and torsional movements easily For this we assume a lateral force $\boldsymbol{F}_{lat}$ of 10 µN applied at a tip of 20 µm and compare the cantilever's lateral displacement and torsional angle as a function of its cross-sectional geometry, i e , we vary the cross section from the square form (15 µm × 15 µm) to a very flat form (4 5 µm × 50 µm) by keeping the area constant (225 µm^2)

The result is shown graphically in Fig 4 where the bending and torsional behaviour of a simple cantilever beam with different aspect ratios is illustrated We see clearly that a flat cantilever tends to react more by torsion (dotted curve) than a symmetrically built cantilever, which enhances the bending movement (solid curve) when the same lateral force is applied

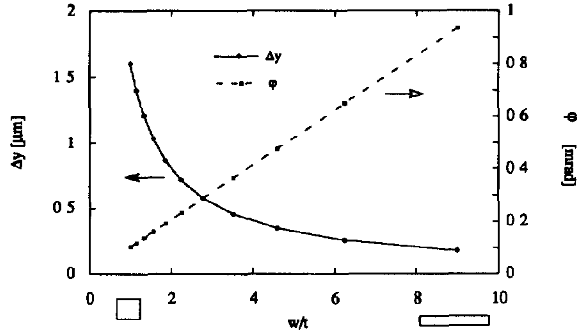

Fig 4 Bending and torsional behaviour as a function of the cantilever geometry (w and t), cantilever length $l = 700$ µm, tip height $r = 20$ µm, lateral force $F = 10$ µN, area $w \times t = 225$ µm^2 = const

Stress evaluation of such free-standing m-Si cantilevers has shown that for a force of 10 µN at the end of the cantilever (typical measurement forces range between tenths of µN and nN [1]), the maximal stress at the clamping ($\sigma_{max} = 15 \times 10^6$ N/m^2) is still about two magnitudes below the fracture stress of silicon [22] It is also very well suited for resonance methods because of its very high intrinsic Q-factor [23]

3. Microfabrication of sharp silicon tips

The crucial part of the AFM point probe is the tip, which ideally should have a curvature radius in the range of some nanometers, an aperture angle of about 5° and a height of about 10 µm, which is crucial for an easy sample approach In the field of vacuum microelectronics both wet and dry etching techniques to form sharp silicon field emitters have been published [24], not all suitable for a full batch integration onto a cantilever, e g , electron-beam deposition inside an SEM [25] or evaporation of a metal through an orifice [26] Sharp pyramidal Si tips can be achieved with a two-stage isotropic–anisotropic etching exploiting the self sharpening due to silicon crystal plane orientation [27] or using reactive ion etching techniques with SiO_2 or Si_3N_4 etch masks [28] by exploiting the underetching of the etch mask by anisotropic etchants

We investigated methods to form tips in m-Si that are compatible with the batch microfabrication of silicon cantilevers Both dry and wet etching allow sharp tips to be formed The best results, however, are obtained by a combination of both, which has been applied to form highly protruding sharp Si tips with a high aspect ratio Instead of using a double-layer mask of SiO_2 and photoresist (PR) [14] to perform the two-stage tip etching, we used here only one single SiO_2 etch mask for the two successive etch steps This is a major process improvement in view of the tip integration onto the cantilever, described hereafter

We patterned a 15 µm × 15 µm square in 1 5 µm thick thermal SiO_2 and anisotropically etched a 15 µm high Si column, using reactive ion etching

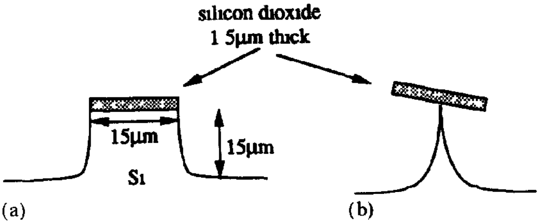

Fig 5 Tip formation exploiting a two-stage dry-anisotropic/wet-isotropic etch process (a) 12 µm high silicon columns obtained by RIE etching (b) Column thinning and pronounced tip formation by wet etching

Fig 6 SEM photograph of a sharp high-aspect-ratio m-Si tip of height ≈ 15 µm

(RIE) with a C_2ClF_5/SF_6 gas mixture With the etch parameters the slope can be greatly influenced [29] Figure 5 shows a rather steep but not completely vertical slope Next, this structure was isotropically etched in a mixture of HNO_3 HF CH_3COOH, which thins the pre-etched shape and forms highly pronounced silicon tips Tip heights up to 20 µm with opening angles of approximately 5 to 10° and tip radii estimated to be 40 nm can be obtained with this technique (see Fig 6) A post process yielding Si tips with curvature radii less than 1 nm by reoxidization and HF etching, exploiting an anomaly of the oxidation behaviour at regions with high geometric curvature, has been demonstrated by Marcus *et al* [30] for other applications

When utilizing very thin high-aspect-ratio tips, the lateral forces occurring during tribological measurements also cause elastic deformation of the tip, i e , the tip will react as a spring itself In return the cantilever would bend less

4. Batch fabrication of silicon cantilevers with integrated tips

The fabrication of cantilevers with integrated tips needs three photolithographies and several dry and wet etching steps, as outlined in Fig 7 Starting from a double-side-polished thermally oxidized silicon substrate, we performed three photolithographic and BHF etching steps in order to obtain on the backside openings for the KOH etching and on the topside a two-step profile SiO_2 etch mask, 0 75 µm thick for the cantilever and 1 5 µm for the tip A membrane, typically 30 µm thick, is anisotropically etched with KOH from the backside while protecting the topside in a mechanical chuck Then the topside is etched with 15 µm deep RIE using chlorine/fluorine gas mixtures to pre-shape the cantilevers An adjustment of the RIE parameters allows vertical sidewalls of the

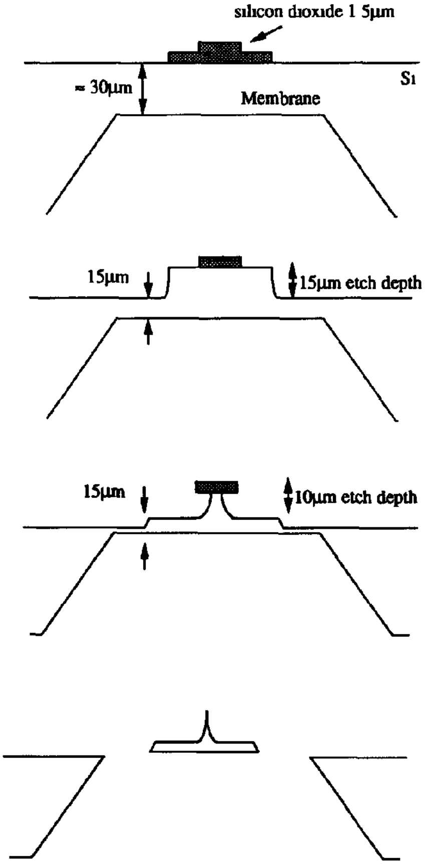

Fig 7 Microfabrication sequence of silicon cantilevers with integrated tips (cf text)

cantilever to be obtained The 0 75 μm thick SiO_2 mask that covered the cantilever is afterwards removed completely and the remaining oxide cap, formerly 1 5 μm and now 0 75 μm thick, serves here as a mask for the tip etching Again RIE is preferred in order to obtain high-aspect-ratio tips and the same process parameters were used as described in the paragraph above SiO_2 withstands well the three successive etch steps (i) 15 μm RIE forming the cantilever shape, (ii) 15 μm RIE etching the silicon column for the tip, (iii) 1–2 min wet etching in HF HNO_3 CH_3COOH sharpening the tip

In accordance with the different requirements for the mechanical behaviour of SFM microprobes, we realized cantilevers with lengths varying from 600 to 1700 μm and widths from 30 to 80 μm which are basically defined by the topside photolithography The cantilever thickness can be varied from ≈5 to 20 μm by the backside KOH membrane etching and the topside RIE etching For 10 μm thick cantilevers, we obtain c_z ranging from 0 5 to 10 N/m and f_0 from 5 to 40 kHz The resonance frequency for one of those cantilevers was measured with a heterodyne optical interferometer and was found to be ≈17 kHz We measured a Q-factor for the cantilever of about 36 000 in vacuum of 10^{-3} mbar

An example of an m-Si cantilever array with an integrated tip, workable only with batch fabrication, is shown in Fig 8 The possibilities of the fabrication process presented here are illustrated in Fig 9, which demonstrates that a tip with a very thin 'supertip' on top of it could be manufactured The AFM head is depicted with an IC underneath it to illustrate a possible application

Fig 9 SEM photograph of a simple beam m-Si cantilever with integrated tip over an IC sample

5. Application

We constructed the holder of the cantilever so that it could easily replace commercial cantilevers in a Nanoscope™ AFM measurement system Since an optical deflection principle is used here, cantilever compatibility was easily achieved It was thus possible to compare the characteristics of the conventional silicon nitride cantilever with our m-Si ones First measurements as shown in Fig 10 display promising results, with about comparable

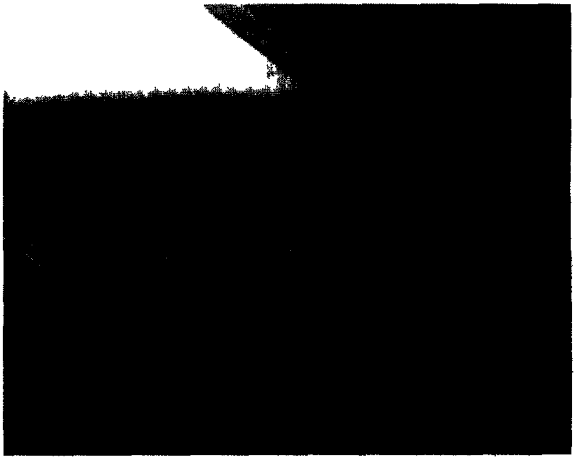

Fig 8 Array of silicon microprobes for multiple measurements

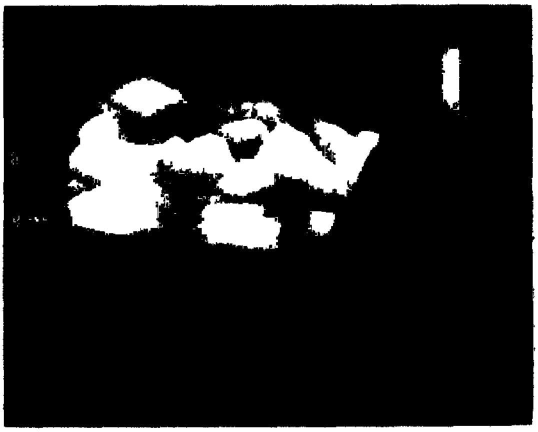

Fig 10 Thin-film diamond surface measured in a Nanoscope™ AFM with one of our integrated silicon cantilever tips

curvatures of the thin-film diamond edges Since such an image is a convolution of the actual topography and the shape of the tip, this is an indication of the usefulness of our tips In these measurements we used a tip shape as in Fig 8 We believe that the sharp tips as shown in Fig 9 will greatly enhance the results

6. Conclusions

Monocrystalline silicon cantilevers with integrated silicon tips could be batch microfabricated using a three-mask process including wet and deep RIE etching techniques A microfabricated solution to allow lateral displacement of an AFM tip that is up to now unique has been demonstrated in this paper Folding the beam gave a more sensitive construction than with a straight beam of the same overall dimensions Also the feasibility of SFM arrays could be demonstrated Moreover, sharp silicon tips with a high aspect ratio, small opening angles and tip radii of only a few tens of nanometres have been fabricated by etching techniques with results similar to those of single-processed tips grown by an electron beam

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Nicolaas F de Rooij received the M S degree in physical chemistry from the State University of Utrecht, The Netherlands, in 1975, and the Ph D degree from Twente University of Technology, The Netherlands, in 1978

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He is a member of the steering committees of the International Conference on Sensors and Actuators and of Eurosensors He acted as European Chairman of Transducers '87 and General Chairman of Transducers '89 He is a member of the editorial boards for the journals *Sensors and Actuators* and *Sensors and Materials*