Piezoresistive scanning probe arrays for operation in liquids

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Abstract. Piezoresistive scanning probe arrays have been developed in view of operation in liquid environments. When the cantilevers are immersed in electrically conductive solutions like for instance physiological buffers, the piezoresistive sensing elements as well as the metal connections have to be passivated. For that purpose, the sensors and the metal wiring were covered with different protective coatings. Long term stability of these passivation layers was demonstrated by imaging in a buffer solution for several hours. Moreover, in view of reducing the damping and thus decreasing the hydrodynamic resistance in liquids, special truss cantilevers have been developed. It was found that this special design conferred no improvement in terms of Q-factor and resonant frequency when operated in water. In order to explain the behaviour of these probes, a theoretical model was established. The model predicted that truss structures could theoretically improve the cantilever performances in liquid, but the probes would need to be operated at high frequency, above 10MHz.

1. Introduction

The scanning probe microscope (SPM) is a versatile tool to measure surface properties with up to atomic resolution in various environments. Its capability to be operated in liquids has led to numerous applications in biology and life sciences [1]. However, a major drawback of SPM is its slow scan speeds and limited scan sizes which limit significantly the throughput. It has been shown that parallel operation of large probe arrays can be used to increase the sensing area and throughput without sacrificing individual sensor sensitivity [2, 3]. Integrated detection of cantilever deflection, e.g. by means of integrated piezoresistive strain gauges [4], simplifies greatly the operation of such probe arrays, but it has to be adapted for liquid environments [5].

In this work we developed various one- and two-dimensional scanning probe arrays with integrated piezoresistive sensors suited for operation in liquids. First, we focused on the passivation of the piezoresistors as well as the metal wiring in order to avoid electro-chemical reactions or leakage...
currents that could occur at the sensors. Second, we investigated special cantilever designs in view of reducing the damping when working in viscous environments.

2. Passivated piezoresistive probe arrays
Different small one- and two-dimensional probe arrays with integrated single crystalline silicon piezoresistors were designed and successfully fabricated by using silicon micromachining techniques. Details about the microfabrication process can be found elsewhere [6].

The scanning electron microscopy image of figure 1 depicts a microfabricated 4x4 probe array. Reference resistors were implemented beneath each cantilever, which made it possible to compensate thermal drift. The tips were about 5µm high and the radii of curvature similar to those of commercially available silicon tips. As the probes were intended to be operated in contact mode on soft biological samples, they were realized with low stiffness. We typically measured spring constants of about 0.1N/m and resonance frequencies around 30kHz.

Figure 1. Scanning electron microscopy (SEM) image of a microfabricated piezoresistive 4x4 scanning probe array. Reference resistors are implemented next to each cantilever.

In order to determine the sensitivity of the piezoresistive sensors, a probe array was mounted in a modified DI Multimode AFM and the resistance change was measured as a function of the cantilever bending. The obtained deflection sensitivity of $1.8 \times 10^{-7} \, \Delta R/R$ per Å is in good agreement with earlier published data for soft cantilevers [7].

The liquid environment of operation imposes stringent requirement on the sensors performances and life-time. Especially when the liquid is electrically conductive or corrosive it is of first importance to passivate the piezoresistors as well as the metal wiring in order to protect them during operation. Our approach consisted of passivating the sensors with a 50nm thin silicon nitride film and the metal lines with a 500nm thick silicon oxide layer. The silicon nitride passivation layer, which covers only the cantilever section where the piezoresistor is implanted, is shown in figure 2a). The silicon oxide film covering the metal connections is also pointed out by arrows on this image.

Several imaging experiments where performed in a phosphate buffered saline (PBS) solution in order to assess the quality of the passivation layers. The pictures of figure 2b) show two atomic force microscopy (AFM) images of fibroblast cells on a silicon substrate acquired in PBS. Both AFM topography images were acquired with the same probe, but at different locations. The cell shown on the left picture is grown on a flat surface area, while the cell imaged on the right picture is trapped inside a groove structure of the silicon substrate. The elapsed time between these measurements was about 6h30min, during which the probe was always immersed in the buffer and operated. These experiments demonstrated the long term integrity of the passivation, since the output signal of the sensors was stable over this period of several hours and no degradation of the image quality could be observed. These experiments clearly demonstrated that the isolation layer sufficiently sealed the piezoresistive sensor and the metal lines against its liquid environment. Moreover, these measurements showed that the fabricated probes were well suited for imaging soft biological material.
3. Piezoresistive truss cantilevers

In liquids the performance of an AFM cantilever in dynamic mode strongly depends on its size and shape [8]. In view of reducing the hydrodynamic resistance in liquids and thus decreasing the damping, cantilevers with reduced face can be used [9]. To that purpose, we investigated special truss cantilevers as depicted in figure 3a). The piezoresistors were, again, incorporated into the short flat section at the fixed end of the cantilever. The passivation of the sensors and the metal connections was realized in the same way than for the probe arrays described earlier. Only the cantilever on the right side in figure 3a) had a reduced face. The one on the left side was solid and used as control lever. The cross-section of the truss part of the cantilever was such that its stiffness was much higher than that of the piezoresistive part. Hence, bending occurred primarily in the latter. A flat paddle was added at the end of the cantilever in order to enable also optical readout.

Figure 3. a) SEM image of a 1x2 piezoresistive truss probe array. Only the cantilever on the right side had an open face. The one on the left side was solid and used as control lever. b) Resonance frequency shift of truss and solid cantilevers when operated in air and resp. in water.
The mechanical responses of the truss and the solid cantilever were tested in air and in water using a piezoelectric oscillator at constant amplitude and frequencies between 2kHz and 50kHz in steps of 0.1kHz. The measured output signal of the piezoresistors is depicted in figure 3b). The Q-factor of the truss cantilever decreased from 83 when oscillated in air to 8.8 when oscillated in water. The solid probe had similar behavior, for which the Q-factor decreased from 58 in air to 8.5 in water. These results show that unlike previously assumed the truss structure does not reduce the damping in water and therefore does not improve the operation of the cantilever under the given conditions. It can also be expected that there is no reduction in thermo-mechanical noise in the cantilever, which is a function of the damping coefficient.

In order to understand why the truss structure did not improve fluid flow through the cantilever and thus decrease its damping coefficient, the geometry was simulated in both 2D and 3D finite element models using ANSYS. The calculations considered the liquid to be incompressible. As the parameter that determines whether the truss structure improves performance, we chose the fluid velocity through the cantilever opening. The geometric parameters indicate that the flow is laminar over the whole range of velocities we are interested in. A critical velocity could be found below which the cantilever acts like a solid beam, i.e. only a negligible amount of fluid goes through the opening. Above this velocity, there is a significant flow through the truss holes. For the given geometry, the critical velocity is 0.173m/s. Moreover, by varying the cantilever thickness, it can be shown that the critical velocity depends on this geometrical parameter. The results of the simulations for three different cantilever thicknesses are shown in figure 4, where the velocity of the fluid inside the truss opening is plotted against the cantilever velocity. The velocity of the fluid has been normalized to the cantilever velocity.

In summary, it is theoretically possible to improve the mechanical behavior of the cantilevers by using truss structures, but a minimum velocity is always necessary. Thus, for the truss structure of the present cantilevers to affect their motion in liquid, the probes would need to be operated at high frequency, i.e. above 10MHz. Furthermore and since the velocity of a cantilever segment is a function of the position along the beam, it is most important to place a truss hole as far to the end of the cantilever as possible. Thus, we would need external excitation with high amplitude and frequency and high resonant frequency cantilevers. The cantilever thickness would be a tradeoff between high resonant frequency and increase in critical velocity.

![Figure 4](image)

**Figure 4.** Graph depicting the normalized fluid velocity in the cantilever opening as a function of the cantilever velocity. It can be seen that there is a transition from negligible flow through the opening to significant flow through the opening.

### 4. Summary and conclusions

Various one- and two-dimensional probe arrays with integrated piezoresistive read-out suited for operation in liquid environments were successfully microfabricated. The passivation of the piezoresistors and of the electrical contacts allowed the probes to be operated in different liquids, such as physiological solutions. The low spring constant of the cantilevers allowed non-destructive contact mode imaging on soft biological material in liquids. In order to assess the long term stability of the passivation, we have cumulated 6h30min of operation in buffer solution without experiencing electrical problems, leakage currents or electrolysis.
Furthermore, we have investigated special truss cantilever with reduced face, which were designed in view of reducing the hydrodynamic force due to the drag during operation in fluids. Experiments performed in air and in water showed that unlike previously assumed the use of this special design conferred no improvement in terms of Q-factor and resonant frequency.

The results of a theoretical study show that a certain minimum cantilever velocity is necessary in order to improve its performance in liquid. There is a critical velocity below which there is nearly no fluid flow through the truss hole and above which we can see significant amount of fluid going through the opening. For the truss structure of the present cantilever, the probe would need to be operated above 10MHz in order that the truss structures affect the probe motion in water. Based on this model optimized cantilever shapes can be established.

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References
[1] Frederix P L T M, Akiyama T, Staufer U, Gerber C, Fotiadis D, Müller D J and Engel A 2003 Curr. Opin. Chem. Biol. 4 641
[2] Minne S C, Adams J D, Yaralioglu G, Manalis S R, Atalar A and Quate C F 1998 Appl. Phys. Lett. 73 1742
[3] Vettiger P, Cross G, Despont M, Drechsler U, Durig U, Gotsmann B, Haberle W, Lantz M A, Rothuizen H E, Stutz R and Binnig G K 2002 IEEE Trans. Nanotechnol. 1 39
[4] Tortonese M, Barret R C and Quate C F 1993 Appl. Phys. Lett. 62 834
[5] Boisen A, Thaysen J, Jensenius H, Hansen O 2000 Ultramicroscopy 82 11
[6] Aeschimann L, Meister A, Akiyama T, Chui B W, Niedermann P, Heinzelmann H, De Rooij N F, Staufer U and Vettiger P 2006 Microelec. Eng. 83 1698
[7] Chui B W, Stowe T D, Kenny T W, Mamin H J, Terris B D and Rugar D 1996 Appl. Phys. Lett. 69 2767
[8] Sader J E 1998 J. Appl. Phys. 84 64
[9] Maali A, Cohen-Bouhacina T, Jai C, Hurth C, Boisgard R, Aimé J, Mariolle D and Bertin F 2006 J. Appl. Phys. 99 024908