# MEASUREMENT OF CHARACTERISTICS OF NANOMETRIC MECHANICAL OSCILLATORS

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## ABSTRACT

For the purpose of making a small probe for scanning force microscopy (SFM), we have succeeded in fabricating nanometric oscillators with a tip mass of silicon and a neck of silicon dioxide by micromachining Si-$SiO_2$-Si laminated substrates. The size of the head ranged from 100 nm to 3 $\mu$m and the diameter of the neck range from 10nm to 200nm. The resonant frequency is calculated to be in MHz order. Results of the test done by an atomic force microscope built within a scanning electron microscope showed that the oscillators are elastic and strong enough for use as SFM cantilevers.

## INTRODUCTION

A mechanical oscillator with a tip on one end is used in non-contact scanning force microscopy (SFM)[1,2] to detect the force gradient acting on the tip, or a mass change of the tip. In most SFMs, the oscillators are silicon fabricated micro cantilevers[4-8]. The minimum detectable force gradient of the oscillator is proportional to $(K)^{1/2}(f_0Q)^{-1/2}$[2,3]. To raise the sensitivity of the oscillator, low spring constant K, high natural frequency $f_0$, and quality factor Q are needed. Since the natural frequency $f_0$ is proportional to $(K/m)^{1/2}$ where m is the mass of the oscillator, the mass also has to be lowered alongside spring constant K, to keep the natural frequency high. To raise Q, minimizing the dimension of the oscillator is favorable since it decreases the amount of intrinsic mechanical loss. Therefore, a small oscillator with a very thin neck is suitable for high resolution SFM.

Recently, we have developed a new type of nanometric oscillator having a mass-spring structure, or a head-neck configuration, by micromachining a silicon-silicon dioxide laminated wafer, namely SOI (silicon on insulator)[9] as shown in Figure 1. The head mass serves as the tip. The head of the oscillator is made of silicon and the size range from 100 nm to 3 $\mu$m. The neck is made of silicon dioxide and the diameter range from 10nm to 200nm and the length from 60nm to 1 $\mu$m. Since the oscillators can be made in high density on the wafer, oscillators with almost the same dimensions can be fabricated in numbers.

In this presentation, we report on the fabrication methods of the oscillators, and the static characteristics of the oscillators obtained from experiments conducted by using an atomic force microscope (AFM) placed within a scanning electron microscope (SEM).

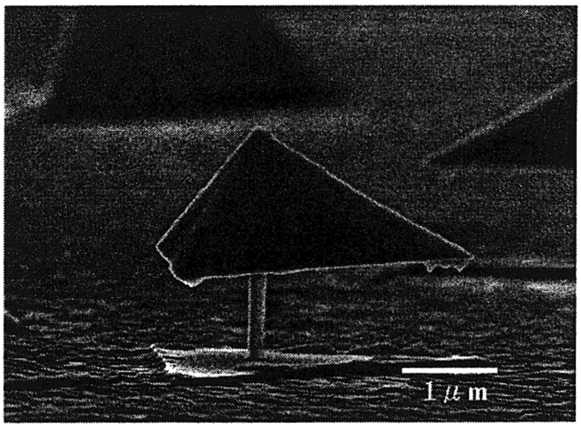



Figure 1: SEM image of a typical nanometric oscillator. The head is made of silicon and the neck is made of silicon dioxide

## FABRICATION

SOI wafers with a silicon-silicon dioxide-silicon layered configuration were chosen for the production of the nanometric oscillators. For fabricating larger oscillators, we have selected bonded SOI wafers as depicted in Figure 2(a). For smaller oscillators, we have selected a kind of SOI wafer called SIMOX (separation by implanted oxygen), which has a silicon dioxide layer made by oxygen implantation (Figure 2(b)). In both cases, the top silicon layer is formed into the head and the silicon dioxide layer is formed into the neck. The different stages of fabrication are depicted in Figures 3(a) to (d) and Figures 4(a) to (e).

We have developed two different methods for structuring the head part. The first method utilizes isotropic etching of silicon by $SF_6$. First, a circular

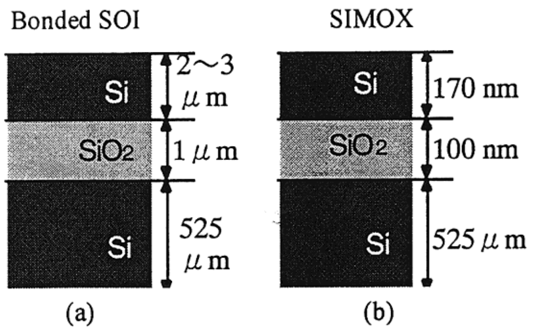



Figure 2: Basic configuration of (a)bonded SOI wafer and (b)SIMOX wafer. The length of the neck of the oscillator is the thickness of the $SiO_2$ layer.

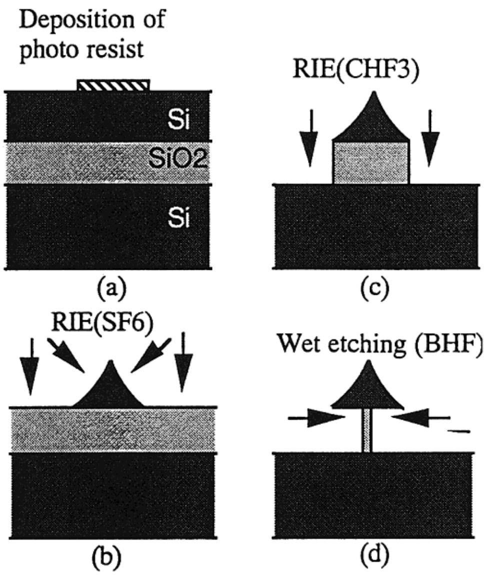



Figure 3: Fabrication process of nanometric oscillator with a conical head by isotropic etching of silicon.

shaped photo resist is deposited on the top silicon layer(a). Then, the silicon is etched isotropically by reactive ion etching (RIE) of $SF_6$(b). A conical head is shaped as shown in Figures 3(a) and (b). The second method utilizes anisotropy of silicon. By combining anisotropic etching by KOH and local oxidization of silicon, the head is shaped to have silicon {1 1 1} surface on two of the sides. As depicted in Figures 4(a) to (c), a tetrahedral head is formed. This method is an existing technique for fabricating quantum dots[10,11]. Both of these methods are applicable to bonded and SIMOX SOI wafers, but for smaller oscillators anisotropic method is favorable since the size of the head is not dependant on etch time or the accuracy of the mask.

The shaping of the neck is done by implementing vertical RIE by $CHF_3$ and wet etching by diluted HF to the silicon dioxide layer. First, the head part is used as a mask for RIE by $CHF_3$ to make a column as depicted in Figure 3(c) and Figure 4(d). Next, the silicon dioxide layer of the column is wet etched by diluted HF until the neck is thinned to desired diameter as shown in Figure 3(d) and Figure 4(e). The length of the neck is determined by the thickness of the silicon dioxide layer.

Figure 5(a) shows an example of an oscillator with a conical head and Figures 5(b) to (d) show examples of oscillators with tetrahedral heads. Since the etching rate of the silicon dioxide neck by HF was constant and isotropic, the dimensions of the column shaped by vertical RIE determines the dimensions of the cross section of the neck. Because of this fact, the neck has a cross sectional shape that is proportional to the bottom shape of the head and is tapered according to the taper created in vertical RIE procedure as shown in Figures 5(a)(c) and (d).

One of the main reasons for the deviation of the neck diameter of the oscillators fabricated on the same

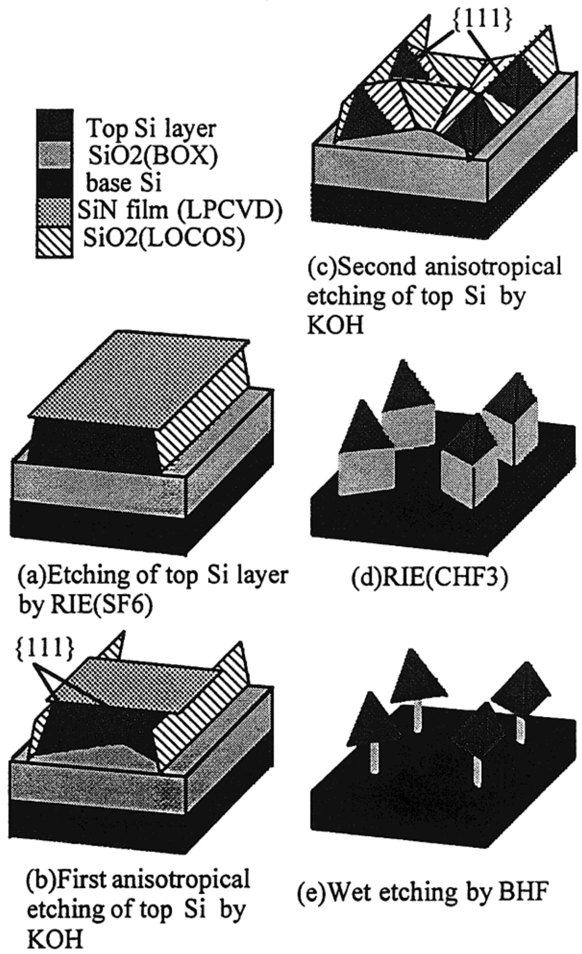
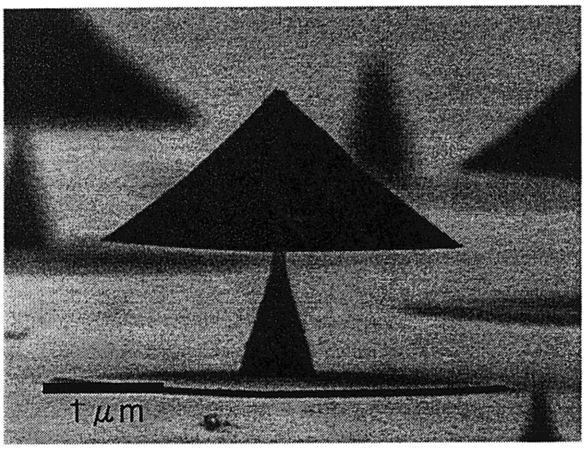
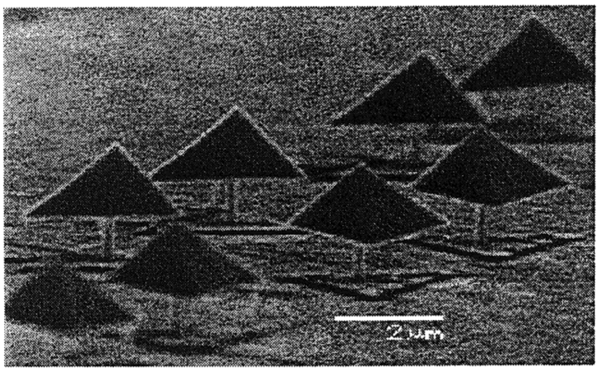


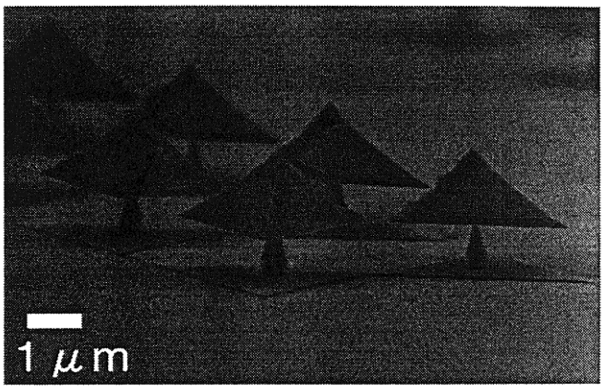

Figure 4: Fabrication process of nanometric oscillator wirh a tetrahedral head by anisotropic etching of silicon.



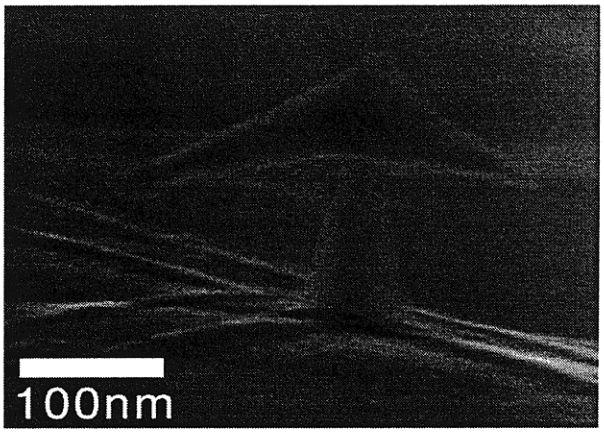

(a) Bonded SOI based oscillator with conical head and tapered neck. The neck diameter is about100nm



(b) Bonded SOI based oscillators with a tetrahedral head and a straight neck. The diameter of the neck is about 120nm.




(c) Bonded SOI based oscillators with a tetrahedral head and a tapered neck. The diameter of the neck is about 120nm.




(d) SIMOX based oscillators with a tetrahedral head and a tapered neck. The diameter of the neck is about 20nm.

Figure 5: SEM images of fabricated nanometric oscillators

wafer is the deviation in the thickness of the top silicon layer. Although the thickness is controlled in high precision, the thickness of the top silicon layer differs among the different regions of the wafer. Since the sizes of the heads are affected by the thickness of the top silicon layer when undergoing the head shaping processes, the diameters of the necks are also affected. As a result, the oscillators fabricated in the same region of the wafer could be made to have almost no visible diversion in neck diameter as seen in Figure 5(b).

## MEASUREMENT OF CHARACTERISTICS

### Instrumentation

We have built an AFM within a SEM. Figure 6 shows the setup of the experiment. A three-degree of freedom coarse positioner with a nanometric resolution is used to press the nanometric oscillator against a conventional AFM cantilever. The actuator used the principle of an inertia drive actuator[17-22]. To pull the oscillator, the tip of the AFM was either glued to the oscillator or made into a hook by depositing hydrocarbon(HC) contaminants within the SEM. This was done by irradiating the electron beam of the SEM on the same spot for few minutes. The amount of deflection of the cantilever was measured from the SEM image. For comparison of size, Figure 7 shows a SOI based tetrahedral oscillator and a conventional AFM cantilever. The size of a SIMOX oscillator is about 10 times smaller than a SOI oscillator.

### Experiments

Figures 8 shows the oscillator with a straight neck being pushed by the AFM cantilever. The picture reveals that the bending is happening almost equally among the length of the neck. The left column of Figure 9 shows the motion of the oscillator when pushed from side and then released. We repeated this

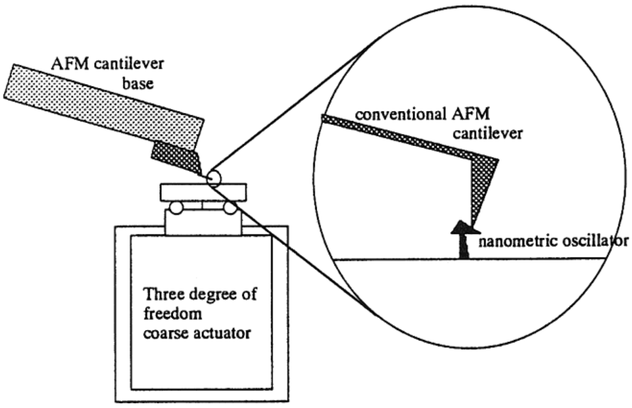



Figure 6: Schematic view of the experimental setup. The three degree of freedom coarse positioner consists of 6 shear force piezos and one tube piezo. The actuator usesthe principle of inertia drive actuator.

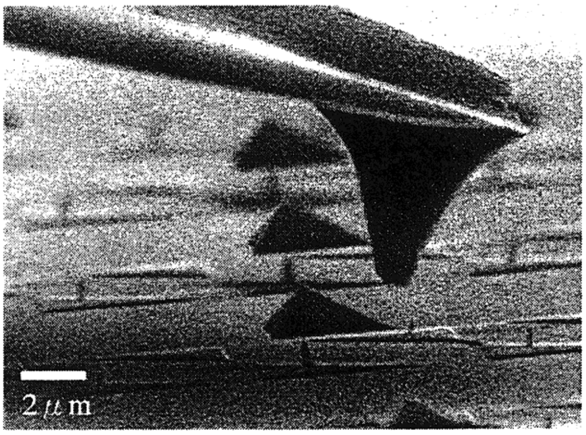



Figure 7: SEM image of a bonded SOI based oscillator and conventional AFM cantilever. The size of a SIMOX based oscillator is about ten times smaller than the oscillator seen in this image.

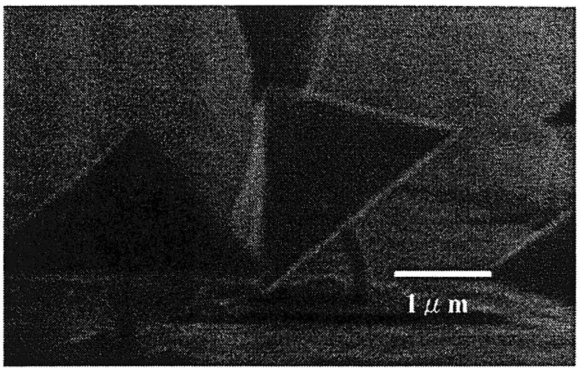



Figure 8: SEM image of a bonded SOI based oscillator being with a straight neck bent. The neck is seen to bend equally among the length.

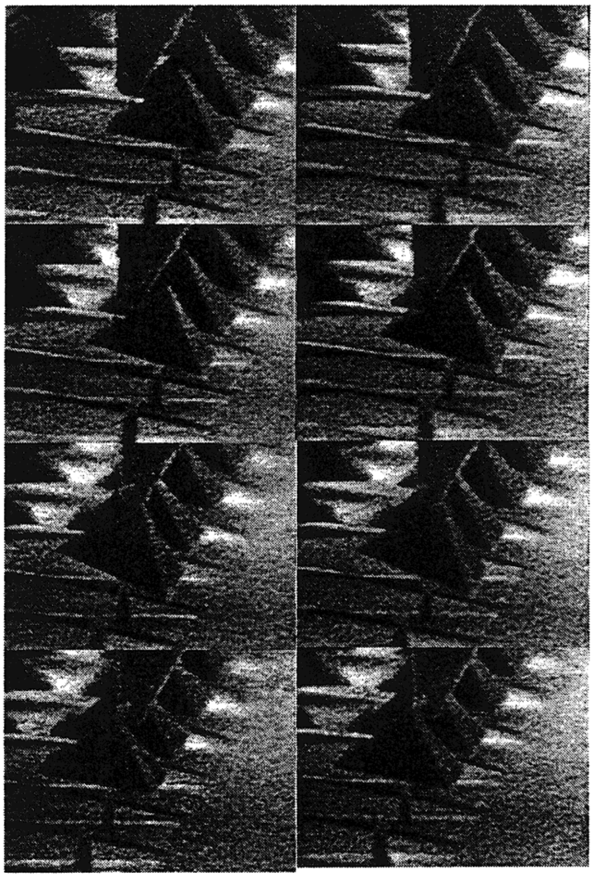


Figure 9: Continuous SEM images of a bonded SOI based oscillator bent for the first time(left column), and bent after 50 times of repetition of the same motion(right column). Note that the motion has not changed. This means that there is no apparent strain hardening nor plastic deformation and that the oscillator is almost perfectly elastic for more than 10 degrees of angular deflection.

motion for nearly 100 times and the oscillator was still not broken. The right column of Figure 9 shows the same motion after 50 times of repetition. Comparing left and right column of Figure 9 reveal that since the motion of the oscillator is the same, the neck shows no strain hardening and thus the bending lies under the elastic limit. The elastic limit of a typical oscillator was seen to be over 10 degrees. When we pushed the oscillator to the point of breakage, the silicon-silicon dioxide interface broke in all of the cases.

To measure the strength of silicon-silicon dioxide interface, we conducted an experiment to measure the force at the point of failure for bending and shear force. In order to measure the maximum bending strength, we anchored the tip of an AFM cantilever to the end of the oscillator and then pulled upward. The oscillator was bent upward until it broke off as shown in Figure 10. In the SEM image of the broken neck, a crack was seen to run diagonally within the neck and not at the interface between silicon and silicon dioxide.

In order to measure the maximum shear strength, we pressed the back of an AFM cantilever against the side of an oscillator with a tapered neck as shown in Figure 11, until the head broke off. The neck was broken at the interface between silicon and silicon dioxide. The amount of force applied to the head at the point of failure measured from the amount of deflection of the AFM cantilever was about $5\times10^{-6}$N. Since the area of silicon and silicon dioxide interface measured from SEM image of the broken neck lies in the order of $10^{-14}m^2$ ($10^4nm^2$), maximum shear stress applicable to the interface lies in the order of $10^8$ N/m$^2$. If we fabricated an oscillator with a neck of about 10nm in diameter, it should still withstand 50nN of lateral force.

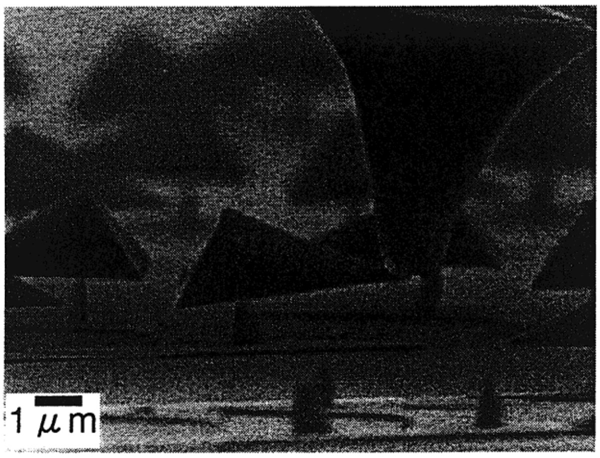



Figure 10: SEM image of an oscillator being pulled upward. The tip of a conventional AFM cantilever is glued on to the head of the oscillator by HC contaminants within the SEM.

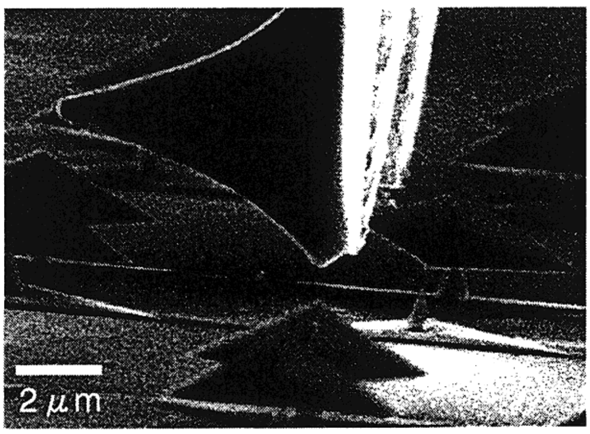



Figure 11: SEM image of an oscillator pushed laterally by the back side of a conventional AFM cantilever.

## FUTURE APPLICATIONS OF OSCILLATORS

Example of a possible configuration for the use of a nanometric oscillator as a SFM probe is shown in Figure 12. For micron sized oscillators, conventional optical lever method can be utilized to measure the deflection of the oscillator. For nanometric oscillators, novel methods have yet to be developed since the oscillator itself lies under the minimum spot size of a laser beam determined by the diffraction limit.

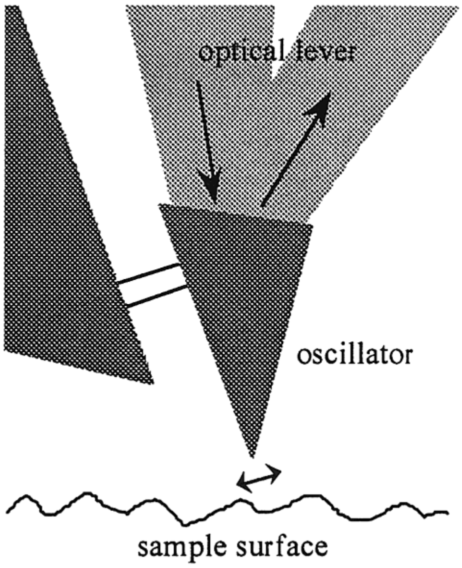



Figure 12: An example of possible configuration of a oscillator as a probe for SFM.

## CONCLUSIONS

We have succeeded in fabricating nanometric oscillators from SOI wafers. The size of the head ranged from 100 nm to 3 μm and the diameter of the neck ranged from 10nm to 200nm. The calculated frequency of the oscillators lies in the range of 10 MHz to 1 GHz and the spring constant in the range of 0.1 N/m to 100 N/m. By using an AFM built within a SEM, we have observed the properties of the oscillators. From experiments, it was found that (i)the bending of the neck occurs equally among the length of the neck in straight necked oscillators, (ii) the neck has an elastic property up to bend of over 10 degrees, (iii) the neck survived repeated stress without any apparent strain hardening for bend of 10 degrees, (iv) the interface between silicon and silicon dioxide can withstand shear stress of $10^8$N/m$^2$ order, which is 50nN of lateral force for a $\phi$ 10nm neck. From the results above, we can conclude that the oscillators are strong and elastic enough to be used as a force detection probe for SFMs, and that further thinning of the neck is possible. Our future studies will focus on the measurement of the dynamic properties of the oscillator to estimate the actual force detection sensitivity of the oscillator when used as a SFM probe. Also, high S/N detection methods are under development to exploit the intrinsically low thermal noise of the oscillator. If a SFM with a nanometric oscillator as a probe is realized, it should show unprecedented sensitivity.

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