Novel Fabrication Method for Surface Micromachined Thin Single-Crystal Silicon Cantilever Beams

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Abstract—This paper describes a novel technique for the fabrication of surface micromachined thin silicon cantilever beams using merged epitaxial lateral overgrowth (MELO) of silicon and chemical-mechanical polishing (CMP). The objective is to demonstrate the feasibility of using this novel technique for the fabrication of arrays of ultrathin, low-stress, single-crystal silicon cantilever beams for use in ultrahigh sensitivity surface-stress or resonant-frequency-based chemical or biological detection schemes. The process flow used in this work will be described in detail and the issues that were faced during the fabrication will be discussed. Cantilever beams with thickness of 0.3–0.5 μm that were $10-25-\mu m$ wide and $75-130-\mu m$ long were fabricated. Mechanical characterization of the cantilever beams were performed by measuring their spring constant using the "added mass" method, which also demonstrated the use of these initial structures to detect masses as low as 10-100 pg. Further work is underway to scale the thickness of these beams down to the sub-100-nm regime.

Index Terms—Cantilever beam, chemical—mechanical polishing (CMP), merged epitaxial lateral overgrowth, silicon-on-insulator (SOI).

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I. INTRODUCTION

N recent years, micro- and nanoscale cantilever beams have become important micromachined structures that have found usage in diverse applications as sensors and actuators. Cantilever beams were first introduced to the nanotechnology field with their use as force sensors in atomic force microscopy (AFM) [1]. They have also been used extensively as probes in various other imaging techniques, involving different interactions between the probe and the sample, that are collectively described as scanning probe microscopy (SPM) [2]. In the

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literature, numerous techniques have been reported which have modified the AFM setup to conduct highly innovative and powerful experiments to measure different phenomenon at the molecular level. An overview of the various areas in which the cantilever beam has been used as a micromechanical sensor can be found in [3].

This paper presents the use of selective epitaxial growth (SEG), epitaxial lateral overgrowth (ELO), and chemical–mechanical polishing (CMP) for the microfabrication of thin single-crystal silicon cantilever beams. This paper presents the details of the fabrication process flow as well as the mechanical characterization of the cantilever beams by measuring their resonance frequencies and spring constants. The present thin cantilever beams can be integrated into silicon-based micro-systems such as flow sensors, pressure sensors, biochemical sensors and the like. The present process can easily be extended to produce low-stress sub–100-nm thickness cantilevers for ultra-high sensitivity chemical and biological detection.

II. CANTILEVER BEAMS WITH NANOSCALE THICKNESS

A. Motivation

The basic impetus for decreasing the dimensions of the cantilever beams is their corresponding increase in the mechanical sensitivity to perturbations from the surroundings. Studies have been published that study the affect of cantilever dimensions on its performance as a force sensor [4], [5], surfaces stress sensor [6], [7] and mass sensor [8]. There will be different design rules for different types of sensing schemes. But generally speaking, decreasing the dimensions of the cantilever beams can improve the performance of the cantilever beams. The thickness of the cantilever beam is of more interest, as it is the hardest to control during fabricating. The thickness is also the dimension that most affects the mechanical sensitivity of cantilever beams [9].

B. Reported Nanoscale Cantilever Fabrication

Single-crystal materials are preferred materials to make sensor elements due to their high mechanical quality factor [10]. Silicon is usually preferred for fabricating sensor elements due to advantages such as low stress and controlled material quality, using currently available VLSI circuit fabrication facilities, miniaturization of devices, high control of dimensions, and the economical advantage of batch fabrication. In addition, if piezoresistive detection modes are preferred, especially due to the need for arrays of cantilevers and detectors, then silicon provides the capability to realize such resistors to detect

deflections. Various methods for the fabrication of ultra-thin cantilever beams have been reported. Among the works that reported fabricating silicon cantilever beams, virtually all of them employ an SOI wafer as the starting material [4], [11]–[13]. Where it has been reported, the SOI wafers used in these processes were obtained from SIMOX (Separation by ion implantation of oxygen) process [11], [12]. Work has also been reported on fabricating nanosized cantilever beams using other materials such as silicon nitride [14] and metals [15].

C. Selective Growth of Silicon

The cantilever beams in this work were fabricated using merged epitaxial lateral overgrowth (MELO) and CMP of silicon. MELO can be regarded as an extension of selective epitaxial growth (SEG) and epitaxial lateral overgrowth (ELO). SEG is a form of vapor phase epitaxy (VPE), and is a variation on the conventional full wafer epitaxy process. Fig. 1 shows a cross-sectional SEM of single crystal silicon grown selectively from a seed-hole region on a silicon wafer. A description of the various forms of selective silicon growth that can accomplished can be found in [16].

The fabrication method described in this work is a novel technique that allows for fabricating all-silicon structures without any oxide layer being present under the silicon anchor of the cantilever beam [17]. This eliminates any mismatch in material properties between the silicon and silicon dioxide material that exists when using SOI as the starting material. The cantilever beams have to be stress free in order to avoid curling of the cantilevers that hampers their proper operation. Residual stresses in cantilever beams are also a source of vibrational energy loss [10]. The present fabrication method has the potential of fabricating arrays of cantilever beams with varying length, width, and thickness dimensions on the same substrate. This can also allow the fabrication of arrays of cantilever beams with a range of mechanical resonant frequencies and sensitivities. In addition, it is also important to note that the techniques described herein lay the ground work for the use of confined lateral epitaxy or tunnel epitaxy to fabricate sub-50-nm-thick cantilevers for single molecule detection applications.

III. THIN CANTILEVER FABRICATION PROCESS USING SELECTIVE SILICON GROWTH

The process begins with growing a layer of silicon dioxide followed by photolithography and buffered hydrofluoric (BHF) wet etching of the oxide to define the cantilever shapes. In order to get the best SEG silicon material quality with the minimum density of defects, it is preferred that the patterns be aligned along the $\langle 100 \rangle$ direction [18]. Another oxidation step is performed to grow a thinner layer in order to obtain the buried oxide layer as shown in Fig. 2(a). The step in the oxide thickness defines the cantilever thickness. (Different cantilever beam thickness can be achieved on the same substrate by performing a series of selective masking, etching and oxidation over the buried oxide layer in order to get different oxide step heights). This is followed by reactive ion etching (RIE) of oxide using CHF₃/O₂

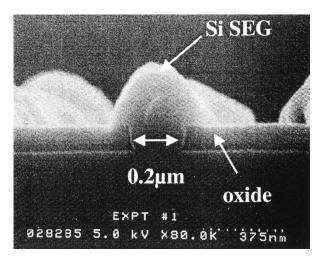


Fig. 1. Scanning electron micrograph (SEM) of single crystal SEG and ELO silicon on (100) surface with patterns aligned to the $\langle 100 \rangle$ direction.

to open the seed windows, as shown in Fig. 2(b), from where silicon is grown epitaxially. A sacrificial oxide is grown and wet etched to annual the damage caused by the RIE step.

The selective epitaxial growth is done in a Gemini I pancake type reactor at T = $970 \, ^{\circ}$ C and P = $40 \, \text{torr using hydrogen}$ (H₂) as the carrier gas, dichlorosilane (DCS) as the source and HCl to maintain selectivity over the oxide. Fig. 2(c) shows the top view and cross-sectional diagram at this step. CMP was performed using a combination of slurries NALCO 2350 (for a faster etch rate) and NALCO 2355 (for finer etching) to etch the overgrown silicon to a flat surface using oxide as an etch stop, as shown in Fig. 2(d). It was necessary to polish the silicon down to the wafer flat surface, otherwise, the silicon forming the anchor of the cantilever beams would have been completely etched away when releasing the cantilever beams, as explained below. A thin layer of oxide (\sim 30 nm) was grown to act as an etch stop against tetra-methyl-ammonium-hydroxide (TMAH) and an etch window was opened in the oxide layer using RIE with CHF_3/O_2 , as shown in Fig. 2(e). TMAH, (25 wt. % in water) at T = 90 °C, was used to etch the surrounding silicon and release the cantilever beams. The wells formed beneath the cantilevers can also be used to form microfluidic channels in the substrate. After etching the surrounding silicon using TMAH and releasing the cantilever beams, as shown in Fig. 2(f), the oxide surrounding the beams was etched away using BHF to result in the final released cantilevers as shown in Fig. 2(g).

Even though the wells underneath the cantilevers were about $14{\text -}16~\mu\text{m}$ deep, the problem of stiction was encountered following each of the above two stated wet etching steps, since the cantilevers were thin $(0.3{\text -}0.5~\mu\text{m})$ and long $(70{\text -}200~\mu\text{m})$. Slightly different stiction prevention steps were performed following each of the two wet etching steps. After the TMAH etch of silicon to release the cantilevers, the structures were rinsed in deionized (DI) water for 10 min, followed by treatment in methanol in three steps of 10 min each. The structures were finally air-dried.

Surface modification by coating with hydrophobic self-assembling monolayers (SAMs) films was used to minimize stiction after the BHF etching of the oxide surrounding the cantilevers. The series of process steps were adapted from [19]. In

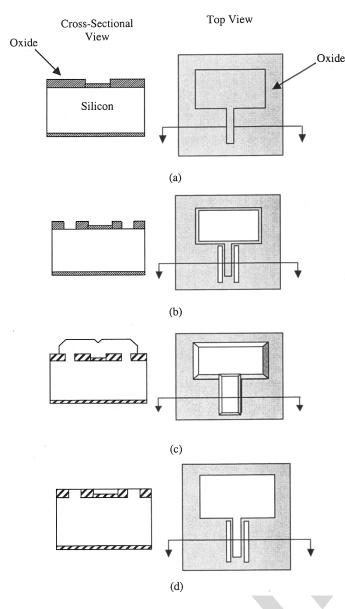


Fig. 2. (a)–(d) Process flow used in the microfabrication of thin cantilever beams using MELO and CMP.

this work, 1.0 mM of octadecyltrichlorosilane (OTS) in 2,2,4-trimethylpentane (isooctane) as the solvent was used to form the SAM coatings. The structures were etched in BHF for 20 min, followed by a 10 min rinse in DI water. The structure was then treated with hot hydrogen peroxide (H_2O_2) for 10 min in order to obtain a very thin oxide layer on the structures. The structures were then rinsed in DI water for 10 min. Following the DI rinse, the structures were rinsed with 2-propanol (isopropyl alcohol) and then with isooctane for 10 min each. The structures were then treated with OTS for 10 min. The structures were then re-rinsed in fresh isooctane, 2-propanol and DI water for 10 min each and finally air-dried.

IV. RESULTS AND DISCUSSION

A. Processing Issues

A total of three masking steps were needed in the fabrication process. In the present work, cantilever beams with thick-

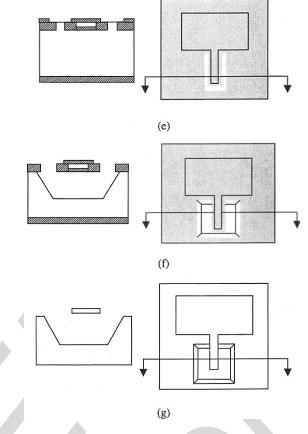


Fig. 2. (Continued.) (e)–(g) Process flow used in the microfabrication of thin cantilever beams using MELO and CMP.

ness of around 0.3–0.5 μ m have been fabricated. A clearance of around 14 μ m was achieved between the cantilevers and the substrate. Prior to the final oxide etch, the cantilevers were found to be bent upwards, as shown in Fig. 3. Once the oxide is completely removed, the silicon cantilevers become flat, indicating no residual stress within them. Fig. (4a) and (b) show scanning electron micrographs of a rectangular and a U-shaped cantilever obtained using this process. Cantilever beams of lengths 78 μ m and 129 μ m, and widths of 13 μ m and 23 μ m, were fabricated and released. The width of cantilevers produced is a function of the distance that the lateral epitaxy has to be grown before it merges from the growth front from the other side. If the selective growth is performed for too long of a time, an undesirable level of nucleation can be produced, resulting in an overall poor material quality. Using the HCl/DCS ratio reported earlier, we limited growth to about 15 μ m so that 20 μ m wide cantilevers could be comfortably produced.

The merging of the growth fronts is also an important factor to consider and optimize. In general, the slope of the sidewall facets decreases with decreasing HCl/DCS ratio [20], which is desirable in order to produce defect-free merged regions. However, as the HCL ratio is decreased, the selectivity of the silicon growth on oxide also decreases, resulting in a worsening of material quality. Data from literature from prior works can provide a process window for achieving high quality merged regions. It should also be pointed out that narrower structures with just one-sided growth could also be formed eliminating any issues with the merged silicon region.

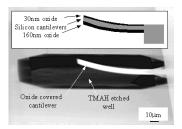
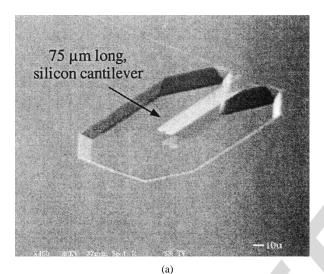


Fig. 3. An angled view of a silicon cantilever beam (prior to the final oxide etch) curling upward. The inset shows the oxide thickness expected from the process used in this work.



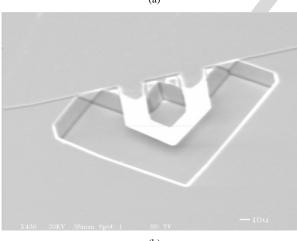


Fig. 4. SEMs of (a) released rectangular shaped cantilever beam and (b) released U-shaped cantilever beam.

B. Mechanical Characterization of Cantilever Beams

The resonant frequency was measured in order to perform the mechanical characterization of the cantilever beams. Thermal mechanical noise is sufficient to oscillate the cantilever beams whose deflections can be detected by an AFM [21]–[24] that employ the optical lever technique [25]. The advantage of this method over driving the cantilever using a piezoelectric is that it does not excite other stiffer, higher mechanical resonance modes such as that of the cantilever holder. In the present set-up, the cantilever deflection signal was extracted from a Dimension 3100 SPM [26], using the DI signal access module, and then

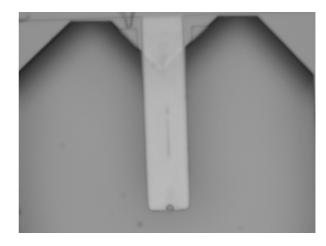


Fig. 5. Top view of cantilever 1 showing spherical polystyrene bead of diameter 3.6 μ m and mass 26 pg attached at the free end of cantilever.

digitized. The power spectral density (PSD) of the signal was then evaluated using MATLAB software. The thermal spectra data was fit to the amplitude response of a simple harmonic oscillator (SHO) [23]

$$A(f) = A_{dc} \frac{f_o^2}{\sqrt{\left[(f_o^2 - f^2)^2 + \frac{f_o^2 f^2}{Q^2} \right]}}$$
 (1)

where f is frequency in hertz, f_o is the resonant frequency, Q is the quality factor, and A_{dc} is the cantilever amplitude at zero frequency. The resonant frequency of a spring system is given as

$$f_o = \frac{1}{2\pi} \sqrt{\frac{k}{m^*}} \tag{2}$$

where k is the spring constant and m^* is the effective mass.

In order to determine the stiffness (or spring) constant of the cantilever beams, the added mass (or Cleveland) method [21] was used. In the present study, polystyrene spherical beads [27] were placed at the ends of the cantilever beams using a micromanipulator (see Fig. 5). Spherical beads of diameter of around 5.48 μ m and 3.18 μ m were used. Using the density of polystyrene of $\rho=1.05\cdot 10^3$ kg/m³, the masses of the beads were calculated to be in the range of 90.5 pg and 17.7 pg, respectively. Due to variation in the diameter of individual beads from the stated specifications of the manufacturer's values, the diameter was measured using an optical microscope. The change in resonant frequency, f₁, due to addition of a single mass, M₁, (see Fig. 6) can be used to calculate the spring constant, k, as well as the effective mass, m*, of the cantilever beam [21]. The spring constant can be evaluated using

$$k = (2\pi)^2 \frac{M_1}{\left(\frac{1}{f_1}\right)^2 - \left(\frac{1}{f_o}\right)^2} \tag{3}$$

while the effective mass can be evaluated using

$$m^* = M_1 \frac{f_1^2}{f_0^2 - f_1^2}. (4)$$

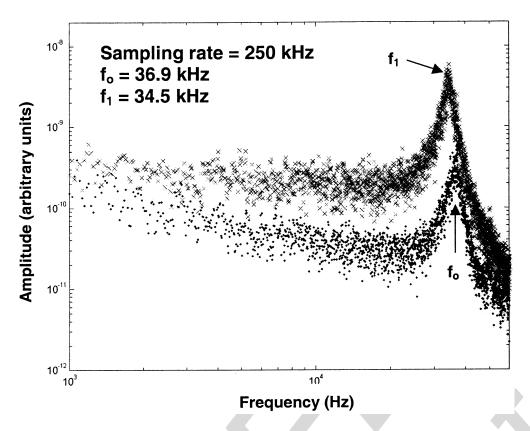


Fig. 6. Resonant frequency change of cantilever 3 after adding a single spherical polystyrene bead of size 5.6 μ m and mass 97 pg, where (\spadesuit) is the unloaded resonant frequency and (\mathbf{x}) is the loaded resonant frequency.

 ${\it TABLE \ I}$ Planar Dimensions of Cantilever Beams With Measured Resonant Frequency and Q

Cantilever Designation	Length, l (μm)	Width, w (μm)	Resonant Frequency (kHz)	Q
1	78	23	85.6	56
2	93	13	70.6	41
3	129	24	36.2	23

Table I shows the planar dimensions of the cantilever beams and their measured resonant frequencies and Q. For one of the cantilevers (cantilever 1), a series of four different masses were added in order to get a linear plot of added mass versus $(2\pi f)^{-2}$. This gives a straight line, as shown in Fig. 7, with the slope giving the spring constant and the y-intercept yielding the effective mass. This can be seen if (2) is rearranged assuming that the masses are added right at the apex of the cantilever beam [21], giving

$$M = k(2\pi f)^{-2} - m^* \tag{5}$$

where, M is the total added mass. For the other cantilever beams only a single mass was added to calculate the spring constant

and effective mass using equations (3) and (4). From the data obtained, it was possible to determine the mass sensitivity of the cantilever beams given as frequency change per unit mass. Table II presents the values of the measured spring constant, effective mass and mass sensitivity for cantilever beams 1 and 3

Table II also presents the values of the extracted thickness of the cantilever beam and the Young's modulus of the silicon material. The planar dimensions of the cantilever can be determined using an optical microscope. Using the planar dimension values and taking density of silicon as $\rho_{\rm Si}=2.33\cdot 10^3~{\rm kg/m^3},$ the thickness of the cantilever can be determined by using the effective mass measured using the added mass method. The Young's modulus can, in turn, be determined from the spring

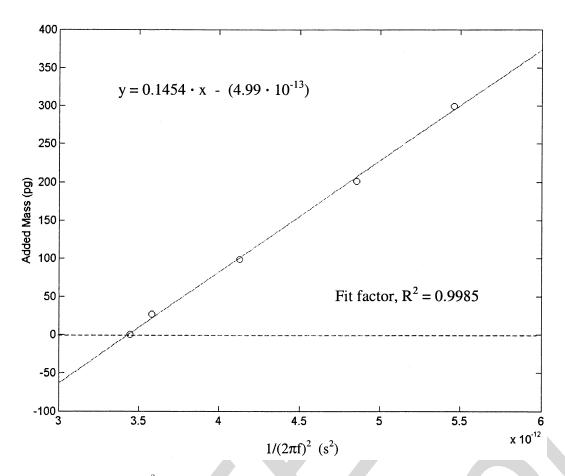


Fig. 7. Linear plot of added mass versus $(2\pi v)^{-2}$ for cantilever 1 with the slope giving the spring constant and y-intercept the effective mass. (Note: The equation displayed in the inset is written assuming that the added mass is in kg).

TABLE II

PLANAR DIMENSIONS AND MEASURED VALUES OF SPRING CONSTANT, EFFECTIVE MASS, AND MASS SENSITIVITY. ALSO LISTED ARE THE
EXTRACTED THICKNESSES FROM THE EFFECTIVE MASS AND THE EXTRACTED YOUNG'S MODULUS FROM THE SPRING CONSTANT
(ASSUMING THE GIVEN PLANAR DIMENSION) FOR CANTILEVER BEAMS 1 AND 3

Cantilever Designation	Length, I Width, w	Measured Spring Constant, k (N/m)	Measured Effective Mass, m* (kg)	Mass Sensitivity (Hz/pg)	Extracted Thickness, t (µm)	Extracted Young's Modulus, E (GPa)
1	$1 = 78 \mu m$ $w = 23 \mu m$	0.145	4.99 · 10 ⁻¹³	65	0.49	100
3	l = 129 μm w = 24 μm	0.0443	9.56 · 10 ⁻¹³	20	0.54	97.4

constant by inserting the planar dimensions and the thickness into the equation for the spring constant of a rectangular shaped cantilever beam given as [28]

$$k = \frac{Et^3w}{4l^3} \tag{6}$$

where E is the Young' modulus, t is the thickness, w is the width, and l is the length of the cantilever beam. From the table, it is seen that Young's modulus of the silicon material is different from that of regular silicon (E = 130 Gpa, [29]). This could

be due to defects that might have been introduced during the growth and merging process of MELO. However, the extracted value of Young's modulus is found to be between 80–110 GPa, and is certainly within the range of the bulk reported value of single-crystal silicon. There was also uncertainty in the thickness values due to the uncertainty that entered in the measurement of the length using an optical microscope, on accounting of the silicon pedestal that was left under the cantilever beams as seen in the SEM micrographs in Fig. 4. This probably caused a layer of oxide to be left beneath the cantilever beam, when

the oxide surrounding the cantilever beam was being etched, causing the effective length of the cantilever beam to be less than that measured using an optical microscope.

V. CONCLUSION

A novel microfabrication technique has been presented that is a viable process for fabricating ultra-thin cantilever beams in single crystal silicon with no stress. Cantilevers with thickness ranging from 0.3 to 0.5 μm , maximum length of around 130 μm and widths of around 20 μm and 10 μm were fabricated using surface micro-machining techniques presented in this paper. Mechanical characterization was performed by measuring the resonance frequency using the thermal noise method and by adding known micro-sized particles. Young's modulus, extracted from the added mass approach was found to be in the range of 80–110 GPa and the mechanical quality factor was measured to be in the range of 20–50 in air. Such cantilevers can also be scaled to thickness of less than 100 nm and can be integrated into micro-fluidic channels within the substrates for a wide variety of chemical and biological detection applications.

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Dr. Bashir received the NSF Career Award for his work in Biosensors and BioMEMS in 2000. He also received the Joel and Spira Outstanding Teaching award from School of ECE at Purdue University, and the Technology Translation Award from the 2001 BioMEMS and Nanobiotechnology World Congress Meeting, Columbus, OH.