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Combined Al-protection and HF-vapor release process for ultrathin single crystal silicon cantilevers

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Abstract

A new technology based on a combination of Al-protection layers and HF-vapor etching to produce ultrathin single crystal silicon cantilevers is presented. 500 μm long, 10 μm wide and 0.5 μm thick cantilevers have been fabricated with a high yield. A resonance frequency of 2 kHz, Q factor >100,000 and a force sensitivity of 6.0×10^{-17} N/Hz$^{1/2}$ have been obtained in vacuum at room temperature for cantilevers annealed at 800 °C.

Keywords: Ultrahigh sensitive cantilevers; HV vapor; Al thin films and MRFM

1. Introduction

Cantilever-based detection of ultrasmall forces is of great interest for several applications in various types of force microscopy. In particular, magnetic resonance force microscopy (MRFM) based on the mechanical detection of magnetic resonance signals in small ensembles of electron or nuclear spins has become increasingly interesting for a large number of researchers. The use of magnetic resonance in combination with scanning tip microscopy was first suggested in 1991 in a theoretical contribution by Sidles, who proposed the mechanical detection of the precession of single spins on a surface [1]. MRFM has been successfully demonstrated for electron spin resonance [2], ferromagnetic resonance [3] and nuclear magnetic resonance [4]. Recently, Rugar et al. [5] has presented the detection of an individual electron spin with an improvement in sensitivity of 10^7 times over the original MRFM setup. MRFM applications require the detection of forces in the attonewton range. This is feasible with ultrahigh sensitive cantilevers with low spring constant ($k < 0.1$ mN/m) and high quality factor ($Q > 30{,}000$). Considerable progress has been made in fabricating attonewton sensitive cantilever [6] but the yield of batch fabrication remains limited [7] due to the fragility of the cantilever during the fabrication. We report here a new fabrication process for highly sensitive single-crystal silicon (Si) cantilevers that lead to a yield higher than 70%. It is based on a combination of two novel process steps for ultra-thin MEMS/NEMS fabrication: (1) protective embedding of the structural Si device in aluminium (Al) during the critical process step of deep dry etching and (2) HF vapor release [8] to avoid the stiction effect usually observed when wet etching is used. The characterization of the realized cantilevers was performed in vacuum using laser beam deflection to measure the resonance frequency and the quality factor. The characterized ultrathin cantilevers showed promising results in vacuum at room temperature.

2. Technology

The starting substrate is a ⟨1 0 0⟩-oriented, 525 μm thick SOI wafer with a 0.5 or 0.34 μm single-crystal silicon membranes and a 0.4 μm buried oxide layer (Fig. 1a). Cantilevers were patterned with photolithography and anisotropic dry etch process with SF_6 at room temperature

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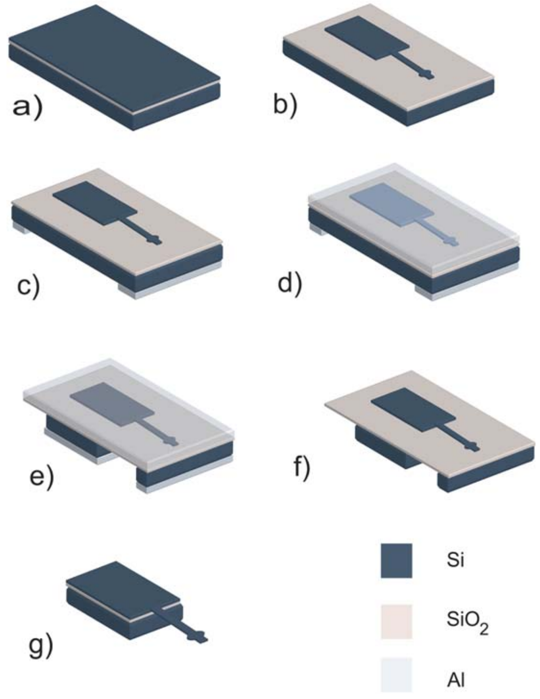

Fig. 1. Schematic illustration of the fabrication process for single crystal silicon cantilever. (a) The starting substrate is a 525 μm SOI wafer with a 0.34 or 0.5 μm thick Si membranes and a 0.4 μm SiO_2. (b) Anisotropic dry etching to structure the cantilevers on the Si membrane. (c) 2 μm-thick Al layer deposited by sputtering and structured by a wet etching. (d) Deposition by sputtering of 2 μm thick Al layer. (e) Deep dry etching of the wafer backside using Al top layer as a mask. (f) Al top and backside layers wet releasing. (g) Oxide releasing with HF vapor.

(Fig. 1b). Two-micrometers-thick Al film was deposited by sputtering on the backside of the wafers. This Al thin film layer patterned with a S1813 positive tone photolithography and Al wet etching at 40 °C (Fig. 1c), acts as a mask for the deep backside etching. After stripping of the photoresist residues with wet remover and oxygen plasma, a 2 μm thick Al layer is deposited also by sputtering on the topside of the wafer (Fig. 1d). This Al layer protects the Si cantilever and reinforces the SiO_2 etch stop layer at the end of the deep dry etching. Anisotropic deep dry etching of the wafer backside is performed in a high inductive coupled plasma reactor (Fig. 1e). The 0.4 μm of buried oxide layer is used as a stop-etching layer. The Al thin films were removed by wet etching at 40 °C (Fig. 1f). Rinsing with DI water of the wafer to clean it from the Al etch solution should be done carefully because of the thin membrane of SiO_2. The latter is etched with a hydrofluoric acid (HF) vapor phase etching system (Fig. 1g), a convenient technique to release fragile, suspended structures and which does not require fluidic water cleaning that can be the reason of cantilever breaking. The wafer to be etched is loaded and fixed onto a chuck. Then the setup is put upside down onto a beaker containing 50% HF in aqueous solution. A feedback system enables to maintain a stable temperature of 35 °C during the etching operation. With an etch rate of 5 μm/h, the 0.4 μm SiO_2 layer was released within 5 min.

3. Results

A SEM picture of one of the realized cantilevers (length $l = 500$ μm, width $w = 10$ μm and thickness $t = 0.50$ μm) with a circular panel for the beam deflection is shown in Fig. 2a. This represents the longest cantilevers realized in this batch. A close-up of the cantilever is represented in Fig. 2b. The fabrication yield reached is approximately 70%. Thirty percent of cantilevers break during the DI water rinsing after the Al wet etching. For 0.34 μm-thick cantilevers the same yield was obtained.

The ring-down method is used to measure the Q factor. The cantilever is excited at its natural resonance frequency with a pulse using a piezoelectric actuator. After the excitation is interrupted, the decay time is measured and the factor Q is deduced from:

$$Q = 2\pi f \times \tau,$$

where f is the resonance frequency of the cantilever and τ the decay time. Several measurements have been carried out in different conditions: air, vacuum, with and without annealing (Table 1). The Q factor in the air at room temperature is below 20. The characterized cantilevers show promising results in vacuum at room temperature. Cantilevers with $l = 500$ μm, $w = 10$ μm, $t = 0.5$ μm, respectively, have $f = 2$ kHz, $k \cong 0.0004$ N/m, and $Q > 100{,}000$. Their force sensitivity, calculated from the expression:

$$F_{\min} = (2kk_{\mathrm{B}}T/\pi Qf)^{1/2}$$

is $6 \times 10^{-17}\ \mathrm{N/Hz^{1/2}}$ (k_{B} is the Boltzmann constant, T the temperature and k the spring constant). The spring

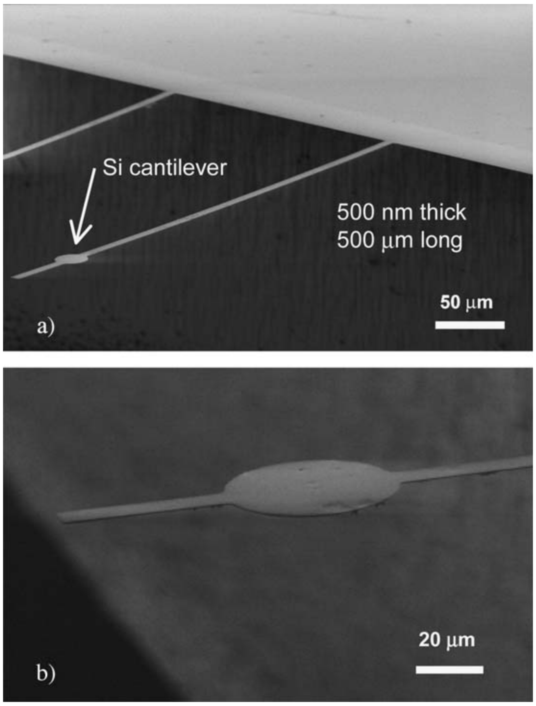

Fig. 2. (a) SEM image of the fabricated single crystal silicon cantilever ($L = 500$ μm, $w = 10$ μm, $t = 0.5$ μm) with the panel for interferometer laser deflection. (b) A close-up of the Si cantilever.

Table 1
Key features of three similar cantilevers with and without annealing at different temperatures

	Not annealed	Annealed at 500 °C 4 h	Annealed at 800 °C 1 h
Cantilever length (μm)		500	
Cantilever width (μm)		10	
Cantilever thickness (μm)		0.5	
Pressure (mbar)		1×10^{-5}	
Temperature (K)		300	
Resonance frequency (kHz)	2.02	2.03	2.03
Spring constant (N/m)		0.0004	
Quality factor	5780	10,470	108,280
Force sensitivity (N/Hz$^{1/2}$)	2.5×10^{-16}	1.9×10^{-16}	6.0×10^{-17}

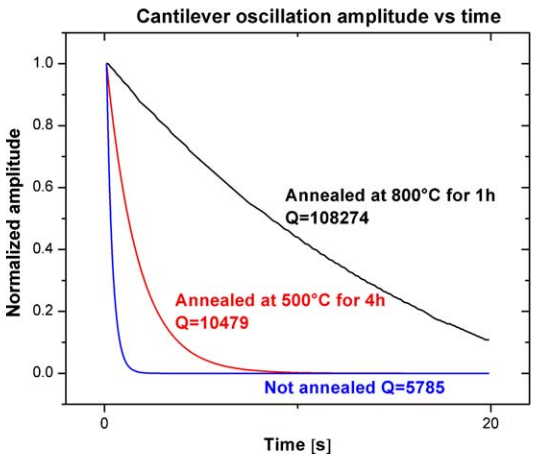

Fig. 3. Ring-down measurement curves for cantilevers with the same dimensions for different annealing conditions. The Q factor increases by a factor of 20 after annealing.

constant is calculated form the expression applicable to the rectangular cantilever that is:

$$k = \frac{E}{4} w(t/l)^3.$$

Fig. 3 shows ring-down measurements at room temperature.

4. Conclusion

The presented technology that combines embedding with Al thin films and the HF vapor releasing is a promising generic method for improving the fabrication yield of fragile freestanding structures. In our contribution, the fabrication high yield is reached for the realization of highly sensitive cantilevers. Annealing allows increasing the Q factor significantly. Due to their high sensitivity the fabricated cantilevers are suitable for MFRM applications.

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References

[1] J.A. Sidles, Appl. Phys. Lett. 58 (1991) 2854.
[2] Z. Zhang, M.L. Roukes, P.C. Hammel, J. Appl. Phys. 80 (1996) 6931.
[3] Z. Zhang, P.C. Hammel, Appl. Phys. Lett. 68 (14) (1996) 2005.
[4] O. Züger, T. Hoen, C.S. Yannoni, D. Rugar, J. Appl. Phys. 79 (4) (1996) 1881.
[5] D. Rugar, R. Budaklan, H.J. Mamun, B.W. Chui, Nature 430 (2004) 329.
[6] B.W. Chui, Y. Hishinuma, R. Budakian, H. Jonathon Mamin, T.W. Kenny, D. Rugar, in: Proceedings of Transducers '03, Boston, 2003, p. 1120.
[7] N.E. Jenkins, L.P. DeFlores, J. Allen, T. Nga Ng, S.R. Garner, S. Kuehn, J.M. Dawlaty, J.A. Marohn, J. Vac. Sci. Technol. B 22 (3) (2004) 909.
[8] T. Overstolz, P.A. Clerc, W. Noell, M. Zickar, N.F. de Rooij, in: Proceedings of the 17th IEEE International Conference on Micro-Electro Mechanical Systems, 2004, p. 717.