

# FABRICATION OF HIGH ASPECT ALUMINUM DOPED ZINC OXIDE NANOMECHANICAL STRUCTURES BY DEEP RIE AND ALD

Nguyen Van Toan\* and Takahito Ono

Graduate School of Engineering, Tohoku University, Sendai 980-8579, JAPAN

\*E-mail: nvtoan@nme.mech.tohoku.ac.jp

## ABSTRACT

This work reports the patterning of high aspect aluminum doped zinc oxide (AZO) for nanowall hollows and capacitive resonators by deep reactive ion etching (deep RIE) and atomic layer deposition (ALD). Nanowall hollows with 50 nm in thickness and 15  $\mu\text{m}$  in height as well as smooth surfaces have been achieved and the aspect ratio of their height-to-width is as high as 300. Suspended AZO capacitive resonators have been successfully fabricated. Its resonant frequency is observed at 10.4 kHz and the quality factor ( $Q$ ) is approximately 500.

## INTRODUCTION

Zinc oxide (ZnO) is a transparent semiconductor material which has wide band gap of 3.3 eV at 300 K and Young's modulus of 150-240 GPa (for thin films) [1]. It is paying attentions for many applications [2-3] such as gas sensors, piezoelectric transducers, and side-gate transistors. There are several methods forming ZnO film such as RF magnetron sputtering [4], chemical vapor deposition [5], pulse laser deposition [6] or ALD [7]. However, for thick film deposition such as several ten micrometers, it is impossible due to low deposition rate. Also, ZnO has some features as follows. The presence of aluminum in ZnO [2] can be significantly improves its electrical conductance. The patterning of ZnO structures can be achieved by both wet and dry etching techniques. High etching rate can be achieved by wet etching [8], but the fabrication of nanomechanical structures meet difficulty due to the lateral etching. In contrast, dry etching using reactive ion etching (RIE) technique [9] can pattern precisely but the achievable aspect ratio is limited by anisotropy and etch selectivity. In such situations, methods for patterning ZnO at micro/nano scale is still an urgent requirement for micro/nano ZnO device fabrication.

This paper presents the fabrication of high aspect ratio AZO structures using deep RIE and ALD. A novel process has been proposed for fabricating nano devices. Nanowall hollow AZO structures and nanomechanical capacitive resonator are demonstrated, respectively.

## HOLLOW AZO STRUCTURES

Hollows can be used for drug delivery and cell transfection. Silicon hollows made of nanowalls of 100 nm thickness with a height-to-width aspect of 16 is presented in [10]. A Si nanobarrel structure with 6.7 nm thickness with an aspect of 50 is demonstrated in [11], but its height is 335 nm. In this work, hollows with a high aspect of 300 and its height of 15  $\mu\text{m}$  are demonstrated. Firstly, silicon pillars with diameter and height of 5  $\mu\text{m}$  and 15  $\mu\text{m}$ , respectively, are formed by deep RIE on a

silicon on insulator (SOI) substrate (Fig. 1(a)). Then, 5% aluminum doped ZnO (AZO) layer with a thickness of 50 nm is deposited by ALD using diethyl zinc (DEZ) and trimethylaluminum (TMA) precursors at chamber temperature of 200°C. AZO film on pillar's surface is sequentially etched by ion beam milling. Finally, silicon pillars are removed by  $\text{SF}_6$  plasma etching. The useful etching property has been found that AZO is not etched during the plasma process. The possible reason is due to the aluminum, which has high selectivity to fluoride gas, consisted in AZO films. The SEM images of the demonstrated nanowall hollows are shown in Figs. 1 (b) and (c).

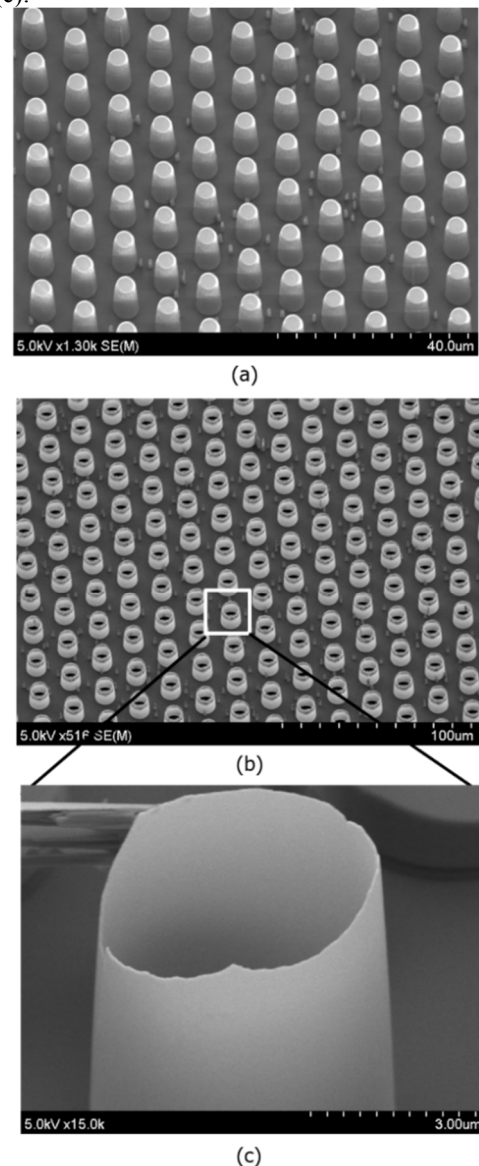


Figure 1. Hollow AZO structures. (a) Silicon pillars. (b) AZO hollows. (c). Close-up image of nanowall AZO hollow.

## AZO CAPACITIVE RESONATORS

Micro/nanomechanical resonators have been studied for a wide range of applications [12-21] such as timing devices [12-15] in oscillation circuits for modern data and communication applications, high sensitive sensors [16-17], and quantum information processing [18], etc. Major advantages are its small mass, high frequency, low intrinsic dissipation and feasibility of integration with IC technology, etc. One of the simplest and most common methods to detect the motion of microfabricated resonators is based on the measurement of the change in capacitance between the sensing electrode and the resonant body [19-21]. However, this method may not be effective for nanomechanical resonators due to its height is in nanoscale. In this work, vertical nanomechanical resonators have been developed to get overcome this problem.

### Device structure and working principle

AZO vertical nanomechanical resonator is shown in Fig. 2. The resonant body is the nano wall hollow cylinder shape, which is sandwiched between driving and sensing electrodes, supported by thin supporting beam on the side and separated by narrow capacitive gap. The resonator is electrostatically excited and vibrated at the resonant frequency as the vibration mode is shown in Fig. 3. When an AC voltage  $V_{AC}$  is applied to the driving electrode, resulting electrostatic force induces flexural vibration mode in the resonant body. Additional DC voltage is also applied to the driving/sensing electrodes in order to amplify the electrostatic force. The resonator is actuated by an electrostatic force which is generated by application of both DC voltage and AC voltage. The output of the resonator relies on the capacitance variation between the fixed electrode and the resonant body. Small changes in the size of the capacitive gap generate a voltage on the sensing electrode.

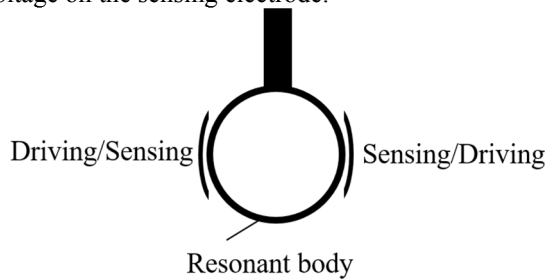


Figure 2. AZO capacitive resonator structure.

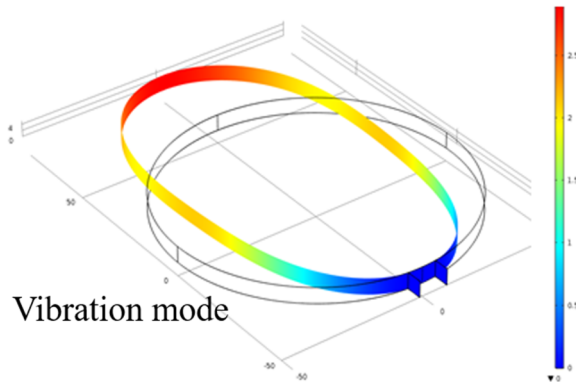


Figure 3. FEM simulation of vibration mode

### Device fabrication

The resonator structures are fabricated by following the fabrication process, as shown in Fig. 4. The details of the fabrication process is as follows.

An SOI wafer which consists of a 15  $\mu\text{m}$ -thick top silicon layer, 1  $\mu\text{m}$ -thick oxide layer and 350  $\mu\text{m}$ -thick silicon handling layer has been employed as a starting material (Fig. 4 (a)). A 500 nm-thick  $\text{SiO}_2$  is deposited by CVD (chemical vapor deposition) on the SOI wafer for the etching mask of the silicon device layer. On this  $\text{SiO}_2$  layer, an electron beam (EB) resist pattern is formed by electron beam (EB) lithography. Then, the  $\text{SiO}_2$  layer is etched by reactive ion etching (RIE). The silicon structures are formed by deep RIE based on Bosch process using  $\text{SF}_6$  and  $\text{C}_4\text{F}_8$  for short etching and passivation cycles (etching time 2.5 s, passivation time 2.5 s) for creating low scallops (Fig. 4 (b)).

Next, the conformal coating of AZO on walls of nano gaps by ALD (Fig. 4 (b)), followed by photolithography and selective silicon etching are performed. Finally, the suspended AZO hollow disk capacitive resonator can be achieved (Fig. 4 (d)).

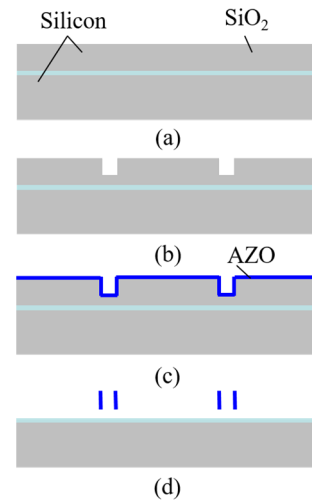


Figure 4. Fabrication process. (a) SOI wafer (15  $\mu\text{m}$  / 1  $\mu\text{m}$  / 350  $\mu\text{m}$ ). (b) Deep RIE with low scallop recipe. (c) AZO deposition by ALD. (d) Plasma etching.

A high aspect ratio of 200 (50 nm-width and 10  $\mu\text{m}$ -height) in the nano resonant body and capacitive gap of 300 nm have been demonstrated, as shown in Fig. 5.

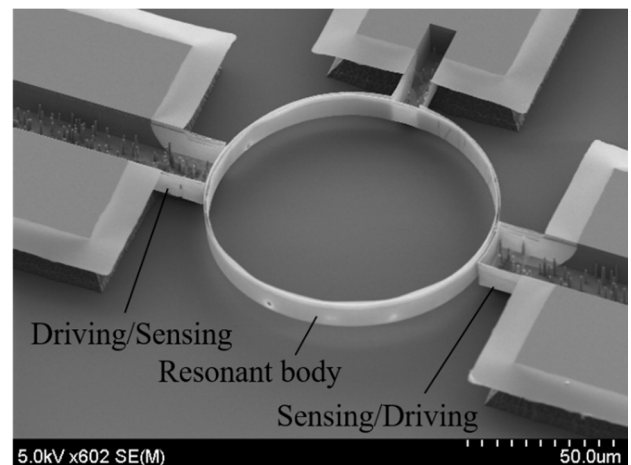


Figure 5. Fabricated AZO capacitive resonator

## Measurement setup and results

A measurement setup and evaluation result are shown in Figs. 6 and 7, respectively. A network analyzer with a frequency range from 10 Hz to 300 MHz has been employed for this evaluation. A DC voltage is applied to the driving and sensing electrodes against the grounded resonator body through a 100 k $\Omega$  resistor, which decoupled from the RF output of the network analyzer using a 100 nF capacitor. The output of the devices is obtained by the capacitive detection between the sensing electrode and the resonant body. Small changes in the capacitive gap generate a voltage on the RF input of the network analyzer. The resonator is placed inside a vacuum chamber at 0.01 Pa with coaxial feed-through.

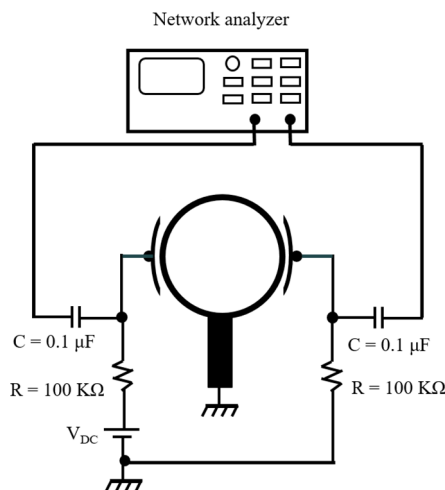


Figure 6. Measurement setup

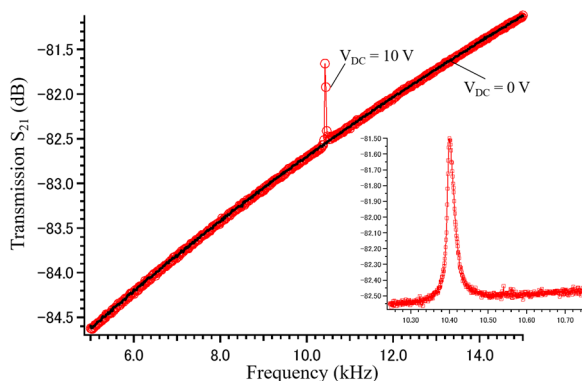


Figure 7. Frequency response of AZO capacitive resonator.

The transmission  $S_{21}$  of the AZO vertical nanomechanical resonator is shown in Fig. 7. A resonant peak, which is observed under measurement conditions  $V_{DC} = 10$  V and  $V_{AC} = 0$  dBm, is found at 10.4 kHz and the  $Q$  factor is approximately 500.

## CONCLUSION

High aspect AZO structures formed by deep RIE and ALD have been successfully demonstrated. Nanowall hollow and capacitive resonator are investigated. This work could be used for many different applications in biomedical engineering, nanomedicine, and life science.

## ACKNOWLEDGEMENTS

The authors thank Mr. Yoshisuke Ansai and Dr. Suguru Sangu for AZO deposition using ALD at Hokkaido University.

Part of this work was performed in the Micro/Nanomachining Research Education Center (MNC) of Tohoku University. This work was supported in part by a Grant-in-Aid for Scientific Research from the Japanese Ministry of Education, Culture, Sports, Science and Technology of Japan, also supported by Special Coordination Funds for Promoting Science and Technology, Formation of Innovation Center for Fusion of Advanced Technologies.

## REFERENCES

- [1] A.V. Desai, M.A. Haque, "Mechanical properties of ZnO nanowires", *Sensor and Actuators A: Physical*, 2007, 134, 169-176.
- [2] Z.L. Wang, "Progress in piezotronics and piezophotonics" *Advance materials*, 2012, 24, 4632-4646.
- [3] A.A.M. Ralib, A.N. Nordin, "Comparative analysis of zinc oxide and aluminium doped ZnO for GHz CMOS MEMS surface acoustic wave resonator", *Proceeding of design, test, intergration and packaging of MEMS/MOEMS (DTIP)*, Barcelona-Spain, 2013, 9, 1-6.
- [4] R.O. Ndong, F.P. Delannoy, A. Boyer, A. Giani and A. Foucaran, "Structural properties of zinc oxide thin films prepared by R.F. magnetron sputtering", *Material science and engineering: B*, 2003, 97, 68-73.
- [5] Z. Chen, K. Shum, T. Salagaj, W. Zhang, K. Strobl, "ZnO thin films synthesized by chemical vapor deposition". *Proceeding of Application and Technology conference*, 2010, 1-6.
- [6] M.G. Tsoutsouva, C.N. Panagopoulos, D. Papadimiriou, I. Fasaki, M. Kopitsas, "ZnO thin films prepared by pulsed laser deposition", *Material science and engineering: B*, 480-483.
- [7] B. Pecz, Z.S. Baji, Z. Labadi, A. Kovacs, "ZnO layer deposited by atomic layer deposition", *Journal of physics: Conference Series*, 2013, 471, 012015.
- [8] A.A.M. Ralib, A.N. Nordin, H. Salleh, R. Othman, "Fabrication of aluminum doped zinc oxide piezoelectric thin film on a silicon substrate for piezoelectric MEMS energy harvesters", *Microsystem technology*, 2012, 18, 1761-1769.
- [9] K.K. Lee, Y. Lou, X. Lu, Peng. Bao. A.M. Song, "Development of reactive ion etching for ZnO based nanodevice", *IEEE transactions on nanotechnology*, 2011, 10, 839-843.
- [10] Y. He, X. Che, L. Que, "A top down fabrication process for vertical hollow silicon nanopillars", *Journal of Microelectromechanical systems*, 2016, 25, 662-667.
- [11] P. Lie, F. Yang, W. Wang, W. Wang, K. Luo, Y. Wang, D. Zhang, "Hard mask free DRIE of

- crystalline si nanobarrel with 6.7 nm wall thickness and 50:1 aspect ratio”, *The 28<sup>th</sup> international conference on micro electro mechanical systems (MEMS 2015)*, Estoril-Portugal, 2015, 77-80.
- [12] J.T.M.V. Beek, R. Puers, “A review of MEMS oscillators for frequency reference and timing applications”, *J. Micromech. Microeng.*, 2012, 22, 013001.
- [13] N.V. Toan, H. Miyashita, M. Toda, Y. Kawai, T. Ono, “Fabrication of an hermetically packaged silicon resonator on LTCC substrate”, *Microsystem Technologies*, 2013, 19, 1165-1175.
- [14] J. Gieseler, L. Novotny, R. Quidant, “Thermal nonlinearities in a nanomechanical oscillator”, *Nature physics*, 2013, 9, 806-810.
- [15] J. Chaste, A. Eichler, J. Moser, G. Ceballos, R. Rurali, A. Bachtold, “A nanomechanical mass sensor with yoctogram resolution”, *Nat. Nanotechnol.*, 2012, 7, 301-304.
- [16] Y.J. Seo, M. Toda, T. Ono, “Si nanowire probe with Nd-Fe-B magnet for attonewton scale force detection”, *J. Micromech. Microeng.*, 2015, 25, 045015.
- [17] N. Inomata, M. Toda, M. Sato, A. Ishijima, T. Ono, “Pico calorimeter for detection of heat produced in an individual brown fat cell”, *Applied physics letter*, 2012, 100, 154104.
- [18] S. Rips, M.J. Hartmann, “Quantum information processing with nanomechanical qubits”, *Phys. Rev. Lett.*, 2013, 111, 049905.
- [19] F.D. Bannon, J.R. Clark, C.T.C. Nguyen, “High Q factor HF micromechanical filters”, *J. Solid-state circuits*, 2000, 35, 512-526.
- [20] R.M.C. Mestron, R.H.B. Fey, K.L. Phan, H. Nijmeijer, “Experimental validation of hardening and softening resonances in a clamped-clamped beam MEMS resonator”, *Proceedings of the eurosensor XXIII conference*, 2009, 812-815.
- [21] N.V. Toan, T. Kubota, H. Sekhar, S. Samukawa, and T. Ono, “Mechanical quality factor enhancement in a silicon micromechanical resonator by low-damage process using neutral beam etching technology”, *J. Micromech. Microeng.*, 2014, 24, 085005.