PAPER • OPEN ACCESS

Novel fabrication of fixed suspended *silicon nitride* structure for MEMS devices with *dry* etching

To cite this article: Khawaja Nizammuddin Subhani et al 2020 IOP Conf. Ser.: Mater. Sci. Eng. 872 012157

View the article online for updates and enhancements.



This content was downloaded from IP address 49.204.231.51 on 09/10/2020 at 07:10

Novel fabrication of fixed suspended *silicon nitride* structure for MEMS devices with dry etching

Khawaja Nizammuddin Subhani^{1,2}, Shubham Khandare¹, R C Biradar² and K N Bhat¹

¹ Centre for Nano Science and Engineering, Indian Institute of Science, Bangalore, India

 2 School of Electronics and Communication Engineering, Reva University, Bangalore, India

E-mail: khawajas@iisc.ac.in

Abstract. A method of fabricating suspended LPCVD and PECVD silicon nitride structure is demonstrated for a wide range of MEMS (Micro-Electro-Mechanical Systems) applications. Low stress LPCVD and PECVD silicon nitride film of 1 μm thickness were selected for the structure separately. Optical Lithographic parameters, viz, photoresist (PR) thickness, PR variety and baking parameters were optimized to obtain the PR suitable for selective etching of silicon and silicon nitride. Parameters of the dry etching process were also optimized to achieve anisotropic etching of silicon nitride and isotropic etching of Si to release the silicon nitride beam. The silicon nitride structures, thus released, were characterized using Scanning Electron Microscope (SEM) and Laser Doppler Vibrometer (LDV). Finite Element Method (FEM) analysis was carried out using COMSOL, to compare with the experimental modes of vibration investigated using the Laser Doppler Vibrometer (LDV). Thus we demonstrate that the first mode at 30 kHz was indeed the optimum match.

1. Introduction

Micro-electromechanical Systems (MEMS) are devices and technologies that have been derived from the microelectronics industry. The construction of self-supporting and suspended structures is one of the fundamental challenges of micro-electromechanical systems (MEMS). Fabrication of most of the MEMS devices use techniques such as bulk or surface micro-machining. The selection of the process will be based on the application.

Silicon nitride material with multiple tweakable properties by varying the parameters of deposition has made it suitable for several applications such as the suspended beam. The suspended structure should have optimum mechanical stress in order to obtain the structure without blister or buckle in the case of highly compressive film and without fracture in case of film having large tensile stress. These are the significant issues confronted in MEMS[1]. The deposition of silicon nitride can be obtained in both Chemical Vapor Deposition(CVD) and Physical Vapor Deposition (PVD) technique. Among the various deposition techniques available, Chemical Vapor Deposition (CVD) is preferred for silicon nitride films due to the lower stress in the CVD film[2]. LPCVD silicon nitrides are mostly preferred compared to PECVD in MEMS applications because of their rigid structures which are hard enough to sustain during the wet etching process [2, 3].

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. Published under licence by IOP Publishing Ltd 1

ICMSMT 2020	IOP Publishing
IOP Conf. Series: Materials Science and Engineering 872 (2020) 012157	doi:10.1088/1757-899X/872/1/012157

Fabrication of suspended silicon nitride structure can be carried out either by wet etching or by dry etching processes. The choice of the etching process is decided based on the ease and productivity of the structure. Usage of wet etching process enables the easy realization of the LPCVD silicon nitride. On the other hand, in the case of PECVD silicon nitride[3, 4], the beam fractures during release[4]. Also, while using the wet etching process for releasing the structure, it is necessary to carry out any of the drying processes such as evaporation drying, sublimation drying, CO_2 supercritical drying, Critical Point Dryer (CPD) release, in order to obtain liquidfree structure [5, 6]. In some cases, it's necessary for extra cleaning steps using HCl in order to remove the residues obtained during the etching on the film^[4] or it requires multiple DI water and IPA rinse for cleaning the residue in some cases [7, 8]. Hence in the existing literature, the focus has been mainly on the properties of the LPCVD and PECVD silicon nitride films and suspended structures were obtained using wet etching technique. However, issues such as the stiction related problems exist in the wet etching technique, which can be solved by multiple additional steps after the release [7, 8]. Also, there are cantilever beam and fixed beam released in dry etching technique[9]. Majority of the process is the combination of dry etching and wet etching for releasing the beam and suspended membrane[10].

The main novelty of this paper is the fabrication of LPCVD and PECVD silicon nitride fixed structures using dry etching technique, which reduces the fabrication processing steps, and at the same time, circumvents the issues such as stiction which is encountered in the wet etching approach. For this purpose, the optimum low-pressure isotropic plasma etching recipe is developed and optimized to release the structure. The release is ensured and characterized using Scanning Electron Microscope (SEM) and Laser Doppler Vibrometer (LDV). Further, the design is validated by simulation in COMSOL using FEM analysis and, also, supported by comparison with the results obtained from LDV measurements.

This type of suspended structure fits in multiple applications. For example, based on the TCR of the Metal, metallized silicon nitride beams can be used for fabricating regular MEMS accelerometer with proof mass[13], thermal accelerometer[14] as well as heater[12]. Also, by understanding the shift in the frequency modes of the Beam, it can be used as a density sensor proposed by some researchers[15].

2. FABRICATION METHODOLOGY

In this work, the fabrication of suspended silicon nitride structure was carried out by the surface micromachining process of silicon nitride deposited on the polished side of a 4 inch < 100 > Si, a wafer of 560 μm thickness. To begin with, RCA wafer cleaning are carried out in order to remove the organic and inorganic contaminant from the wafer followed by dilute HF dip to remove any native oxide present on the Si-substrate. Wafer was next rinsed with DI water and blown dry with Nitrogen. Following the above Si-wafer cleaning process, further process were done as follows:

- (i) On Si-wafer-1 the First nano LPCVD silicon nitride of thickness 1 μm was deposited using DCS (H_2SiCl_2) and NH_3 as the reactant gases with a ratio of 2 : 1, maintaining the pressure inside the furnace at 200 mTorr and the temperature at 850 °C.
- (ii) On Si-wafer-2, PECVD silicon nitride $(1 \ \mu m \text{ thickness})$ was deposited using Plasmalab100 Oxford tool, using SiH_4 , NH_3 and N_2 with a flow of 20 sccm, 20 sccm and 980 sccm at a pressure of 1 Torr and RF power 20 W and substrate temperature at 350 °C.

Both the wafers were separately analysed for estimating the stress induced due to the deposited film.

The individual die was obtained, by dividing the above two sets of wafers into $1cm \times 1cm$ chips, as shown in Figure 1(b), for further processes: the individual die and further processing.



Figure 1. Cross-section schematic diagram of the fabrication process flow.

One of the crucial steps lies in the selection of patterned masks based on the selectivity of the mask material with etch material. The most accessible and more straightforward way is choosing photoresist as mask. The selectivity of (i) silicon nitride to PR (Photoresist) is 1:0.7, (ii) Si to PR is 1:0.07 and (iii) Si to silicon nitride is 1:0.015 which were determined by prior separate experiments. Based on the selectivity, the PR thickness is adjusted by varying the spin coating parameter in order to release the structure. The Details of the process for patterning and releasing the Si_3N_4 structure are as follows:

- (i) For patterning, the design on Si/silicon nitride substrate dies, the optical photolithography process is chosen with AZ5214E positive photo-resist and exposure is done using the lithography tool (MJB4). The pattern was developed using MF26A developer followed by hard baking at 110°C to harden the resist for the post process, as shown in Figure 1(c).
- (ii) The vital step of dry etching, which involves plasma, was used to achieve the efficient suspended released silicon nitride structure. The load-locked Oxford Plasma ICP RIE etching system is employed. Initially, anisotropically etching of silicon nitride films was carried out, followed by isotropic etching of silicon to obtain the suspended structure. The vacuum of 7×10^{-8} Torr was maintained in the chamber of the ICP RIE tool. The ICP RIE chamber consists of ICP of 2 MHz and a substrate RF bias of 13.56 MHz.

The Si-wafer-1 which had LPCVD nitride and those Si-wafer-2 which had PECVD nitride were separately etched anisotropically in ICP chamber at a gas flow rate of 45 sccm SF_6 with ICP and RF power of 2000 W and 150 W and process pressure of 10 mTorr to achieve obtain the LPCVD silicon nitride etch rate of 550 nm/min and the PECVD silicon nitride etch rate of 850 nm/min respectively. An over-etch of 20% was carried out after landing on silicon, as shown in Figure 1(d). For the next step, isotropic dry etching is employed to etch the silicon isotropically and release the silicon nitride structure completely as shown in Figure 1(d). The structure is carefully designed with two fixed anchors to obtain a free-standing silicon nitride beam. Isotropic Si-etching was carried out after anisotropic etching of silicon nitride with ICP chamber maintained with a gas flow of 100 sccm SF_6 gas, ICP and RF power of 800 W and 50 W with process pressure of 7.5 mTorr to achieve lateral to vertical etch rate of silicon layer in the ratio 1:2, as shown in Figure 1(e). This is followed by photo-resist removal with oxygen plasma, as shown in Figure 1(f).

3. RESULTS AND DISCUSSIONS

Experimental characterization plays a vital role in confirming whether the structure is released without affecting the structure dimensions. In order to confirm all the details of the beam, the detailed studies and analysis were carried out using (i) Ellipsometer, (ii) Scanning Electron Microscope (SEM), (iii) KMOS Ultrascan and (iv) Laser Doppler Vibrometer (LDV) were used. The thickness of the (a) LPCVD Low-stress silicon nitride film and (b) the PECVD silicon nitride were both measured to be 1 μm . by using J A Woolem Ellipsometer and the stress of the film was measured about $200 \ MPa$ and $180 \ MPa$ tensile using KMOS Ultrascan as presented in the Table-1

Table 1. Properties Comparison data with LPCVD and PECVD deposited silicon nitride film.

Deposition Technique	Deposition Tempera- ture (^{o}C)	Refractive index (n)	Stress (MPa)	Etch Rate (nm/min)	Selectivity (Silicon nitride:PR)
LPCVD PECVD	$850^{o}C$ $350^{o}C$	$1.99 \\ 2.03$	200 180	550 850	$1:1.09 \\ 1:0.75$

With the directional plasma, the silicon nitride was etched anisotropically an etch rate of $550 \ nm/min$ for LPCVD silicon nitride and $850 \ nm/min$ for PECVD silicon nitride for 140 seconds. To release the structure, isotropic silicon etch was carried out with a lateral etch rate of 2 $\mu m/min$ and a Vertical etch rate of 4 $\mu m/min$ for 170 seconds. Both were carried out in the ICP RIE tool. Figure 2 & 3 show the SEM images of over etching and under etching of the structures obtained while releasing the structure and optimizing the selectivity of the photoresist with silicon nitride and silicon. Figure 4 shows the SEM image of the silicon nitride structure suspended in the air.



Figure 2. SEM image of Over etched sample



Figure 3. SEM image of Under etched sample.



Figure 4. SEM image of suspended silicon nitride beams with Optimized Etching, clearly showing the structure is in the air with two fixed pads.

Laser doppler vibrometer(LDV)[10, 11] is used for high positional accuracy non-contact dynamic displacements. Mode shapes are generally established by taking multiple measurements using single point LDVs. Since it is challenging to measure input forces directly, the excitation voltage is commonly used as a reference for measurement. In this approach, the device is excited by the external piezo-transducers and response measurements is sequentially taken at several points, and then combined to provide mode shapes The frequency response of the device is observed by performing frequency sweep from 10 kHz to 350 kHz by giving external excitation with the input voltage of 1 V.



Figure 5. Comparison of Frequency Vs. Displacement data and First mode of vibration of suspended silicon nitride beams with Experimental LDV data and COMSOL data.

After analyzing data obtained from SEM imaging, the LDV data provided further insight into the intact release structure even after external excitation of 1 V from the piezotransducer. The data collected from the LDV was then verified using the COMSOL Multiphysics which is shown in Figure 5. The resulting condition of a thin film obtained after the fabrication was incorporated into the solid mechanics module of the COMSOL and the eigenmodes of the structure were obtained analytically. The optimal match was obtained for the breathing mode, i.e., the first mode of vibration of the structure found from LDV and COMSOL data was suggestive of the reliability of the fabrication process.

Table 2. Comparison of simulated and LDV data of LPCVD & PECVD deposited film

Data	LDV Measured Data	
LPCVD PECV.	D	
Natural Frequency (kHz) 30.043 29.273 28.457		

Further COMSOL simulation using FEM analysis was carried out to compare the natural frequency obtained by COMSOL simulation and LDV data (shown in Figure 5).

The comparison data in Table 2 shows optimum match at around 30 kHz for LPCVD and COMSOL data whereas 28.457 kHz with PECVD data, which is the first mode of the structure.

4. CONCLUSION

Dry etching is an optimum technique to release narrow structures with the narrow gap by varying process parameters to achieve low of over - etch with the least possible undercut. It may be noted that issues such as stiction, residuals, etc. existing in the wet etching are absent in dry etching methods. In this work, the LPCVD and PECVD silicon nitride beams are successfully released and fabricated using the ICP RIE. The fabricated structure was simulated with FEM analysis in COMSOL to verify the modes of vibration at a different frequency, and an optimum match was found around 30 kHz frequency along with vibrometer data from the micro system analyzer of Fabricated structure. The released beams are confirmed by data obtained from micro system analyzer (LDV) and Scanning Electron Microscope (SEM).

Acknowledgment

We acknowledge funding support from MHRD, MeitY and DST Nano Mission through NNetRA. The authors would also like to acknowledge National Nano Fabrication Center (NNFC) and Micro-Nano Characterization Facility (MNCF) at Centre for Nano-Science Engineering (CeNSE), Indian Institute of Science, Bangalore.

References

- Mackenzie K D, D J Johnson, M W DeVre, R J Westerman, and B H Reelfs 2005 Proceedings of the 207th Electrochemical Society Meeting 207 148-159
- [2] Gaspar Joao, Marek Schmidt, Jochen Held, and Oliver Paul 2007 Reliability of MEMS materials: Mechanical characterization of thin-films using the wafer scale bulge test and improved microtensile techniques MRS Online Proceedings Library Archive 1052
- [3] Stoffel A, A Kovacs, W Kronast, and B Müller 1996 Journal of Micromechanics and Microengineering 6 1-13

- [4] Shi Shuai, Xuefang Wang, Chunlin Xu, Jiaojiao Yuan, Jing Fang, Shengwei Jiang, and Sheng Liu 2013 14th International Conference on Electronic Packaging Technology 14th 23-26
- [5] Kim, Chang-Jin, John Y. Kim, and BalajiSridharan 1998 Sensors and Actuators A: Physical 64 1 17-26
- [6] Jafri, Ijaz H, Heinz Busta, and Steven T Walsh 1999 International Society for Optics and Photonics 3880 51-58
- [7] Tas, Niels, TonnySonnenberg, Henri Jansen, Rob Legtenberg, and MikoElwenspoek 1996 Journal of Micromechanics and Microengineering 6 385-397
- [8] Legtenberg, Rob, Harrie AC Tilmans, Job Elders, and MikoElwenspoek 1994 Sensors and actuators A: Physical 43 230-238
- [9] Linder C, L Paratte, M-A Grétillat, V P Jaecklin, and N F De Rooij 1992 Journal of Micromechanics and Microengineering 2 122
- [10] Kim G M, S Kawai, M Nagashio, H Kawakatsu, and J Brugger 2004 Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures Processing, Measurement, and Phenomena 22 1658-1661
- [11] Ozdoganlar O Burak, Bruce D Hansche, and Thomas G Carne 2005 Experimental mechanics 45 498-506
- [12] Jakoby, Bernhard, Franz Peter Klinger, and Peter Svasek 2005 Sensors and Actuators A: Physical 123 274-280
- [13] Konishi Toshifumi, Daisuke Yamane, Takaaki Matsushima, GhouMotohashi, Ken Kagaya, Hiroyuki Ito, Noboru Ishihara, Hiroshi Toshiyoshi, Katsuyuki Machida, and Kazuya Masu 2013 Japanese Journal of Applied Physics 52 06GL04
- [14] Alrowais, Hommood, Patrick Getz, Min-gu Kim, Jin-Jyh Su, and Oliver Brand 2016 IEEE 29th International Conference on Micro Electro Mechanical Systems (MEMS) 29th 761-764
- [15] Roy, Kaustav, Harshvardhan Gupta, VijayendraShastri, Ajay Dangi, and RudraPratap 2018 Fluid Density Sensing Using PMUTs IEEE SENSORS 207 1-4