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Test Structure for Measuring the Selectivity in XeF_2 and HF Vapour Etch Processes

Markus Rondé, Anthony J. Walton, Jonathan G. Terry

***Abstract*— Etch selectivity between layers is an important parameter in the fabrication of microelectronics and microsystems. This is particularly true in the case of isotropic gas/vapour etching methods used to release free standing structures through the selective etching of sacrificial layers. Commonly used structural materials have been reported to be largely inert when exposed to a given vapour etchant, indicating high selectivity when measured against typical sacrificial layers. However, there is growing evidence that these structural layers are actually etched at an enhanced rate if they are located in the proximity of the sacrificial layer being removed. Hence, removal rates given in the literature, which have resulted from measurements of layers that have been etched in isolation, can no longer be trusted to characterize critical etch processes in device fabrication. In this paper, a test structure is reported that enables a far more appropriate determination of the etch selectivity between sacrificial and structural materials. The method is demonstrated with the two most common vapour etch processes. Firstly, the XeF_2 vapour etch of a polysilicon sacrificial layer located above a silicon nitride structural layer, and secondly, the HF vapour etch of silicon dioxide placed above a silicon nitride structural layer. Both test structure datasets are presented. The polysilicon and silicon nitride layers, etched with XeF_2 show a selectivity of 5:4. The silicon dioxide and silicon nitride layers etched with HF, show a selectivity of 6: 1 to 8: 1.**

***Index Terms*—MEMS, NEMS, Vapour etching, test structure, XeF_2, Hydrogen fluoride**

I. Introduction

Etching sacrificial layers to release free standing structures is a critical process step in the fabrication of micro and nano-electromechanical systems (MEMS/NEMS). Historically, sacrificial layers such as silicon or silicon dioxide were removed using wet etch processes. However, the commercial advantages and novel scientific possibilities inherent with smaller sensors and actuators has fueled a trend towards miniaturization. At the micro and nanoscale, the high surface-to-volume ratios may cause stiction resulting in the adherence of the released structures to the surface underlying the sacrificial layer. Stiction [1] [2] results from surface tension during the drying of the liquid etchant [3] and can be avoided by the use of a gas/vapour phase etchant. Vapour etching of silicon dioxide and silicon by hydrogen fluoride (HF) and xenon difluoride (XeF_2) respectively are now commonly used release processes in industry.

Various test structures have been used to characterize vapour etch processes. For instance, aperture test structures have been

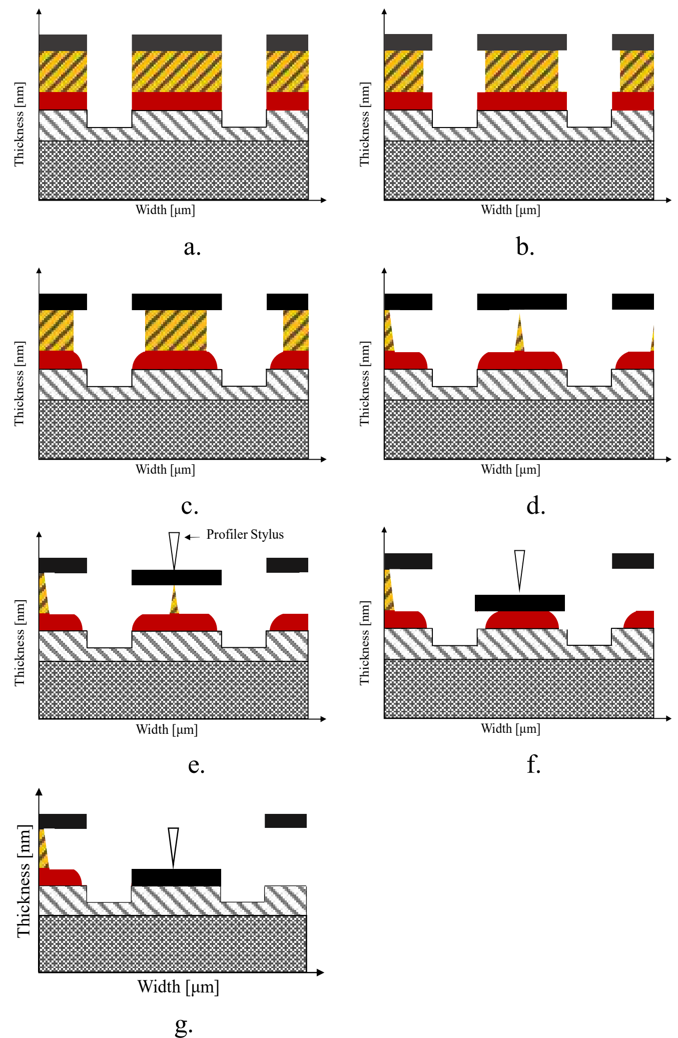

Fig. 1. Shematic of the bridge array methodolgy. (a) shows the test structure before etching. (b)-(c) show the etch initiation on the sacrificial and structural material respectively. (d)-(f) show the test structure with increasing etch time.

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used to measure etch rates, trenching and loading [4][5][6][7]. Sugano et al. [8][9][10] used wagon wheel patterns to accurately measure etch undercuts and to investigate the aperture size effects, surface roughness' and etch rates in XeF_2 etching. Finally, cantilever test structures have been used to demonstrate stiction free etching [11][12] and as proof of concept for novel vapour etch techniques [13].

To date, most vapour etch selectivity data has been obtained by blanket etching of wafers or chips. Blanket layer etching does not reflect the reality of MEMS device manufacturing, because in most applications of sacrificial vapour phase etch processes, the structural layers of a device being fabricated are in close proximity to the sacrificial materials being removed. It has been observed, that a structural material, which is not significantly affected when introduced to the vapour etch in isolation, can be severely attacked when in the proximity of a sacrificial material being etched [14][15]. In this situation, determining the etch selectivity between the materials is non-trivial and requires a measurement method that can quantify the undercut etch rates of two materials on a single die when isotropically etched in close proximity to one another.

Our previous work [16], presented a test structure specifically developed for this purpose along with experimental data for XeF_2 vapour etching. This ICMTS Special Issue paper is an expansion of the conference paper. It includes the original concept, methodology and experimental data and in addition, presents the test structure, fabrication methodology and experimental data for HF vapour etching. This represents test structures and selectivity measurement methodologies for the two most common vapour etch techniques used in MEMS fabrication.

For the XeF_2 experiment, the etch selectivity between a structural plasma enhanced chemical vapour deposited (PECVD) silicon nitride layer and a low pressure chemical vapour deposited (LPCVD) sacrificial polysilicon layer being processed in a commercial xenon difluoride vapour etch tool is determined. Similarly for the HF experiment, the etch selectivities between a PECVD silicon dioxide sacrificial layer and a PECVD silicon nitride layer are measured after processing in a commercial HF vapour etch tool. The following sections briefly explain the measurement method, elaborating on the layout and the design. The interpretation of the resulting surface

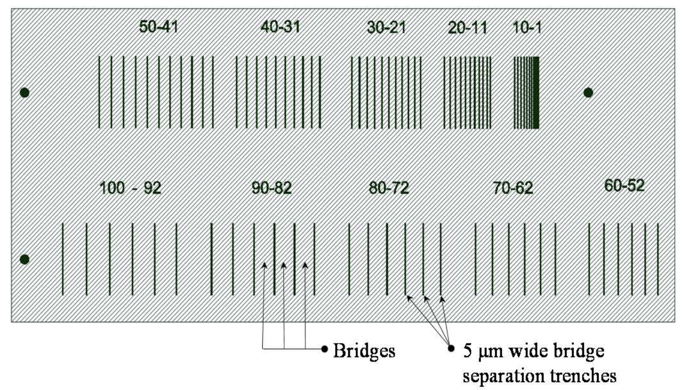

Fig. 2. Design layout of the test structure showing the 5 x 300 µm trenches. Each section is labelled with the bridge width. The trenches are the dark lines and the separation gradually decrease by 1 µm at the top row and 2 µm at the bottom array.

profile is then discussed with the presentation of evidence verifying the characterisation method. Finally a review of the test structures' performance is given using a dataset obtained during experiments. Special attention is given to the post-etch treatment of the HF test structure, as it was found to be important that residual stress in the copper capping layer was optimised for successful operation of the structure.

II. Measurement Method

The test procedure measures the mechanical displacement of an array of etch-released free-standing bridge structures. The width of the bridge structures in the array is incrementally increased in a similar manner to [17] and [18], which uses cantilevers to measure etch rates. Following an etch release process, a profilometer is scanned across the array of bridges and, if release has been achieved, the down force of the stylus vertically displaces the bridges, with the vertical displacement set by the thickness of etched material. The width of the widest bridge structure that has been displaced by the thickness of the sacrificial layer is equal to twice the undercut distance. This undercut distance and the etch time can then be used to determine the apparent etch rate. The structural material is the layer below the sacrificial material and the etch rate of this second material can be obtained in a similar manner, from the

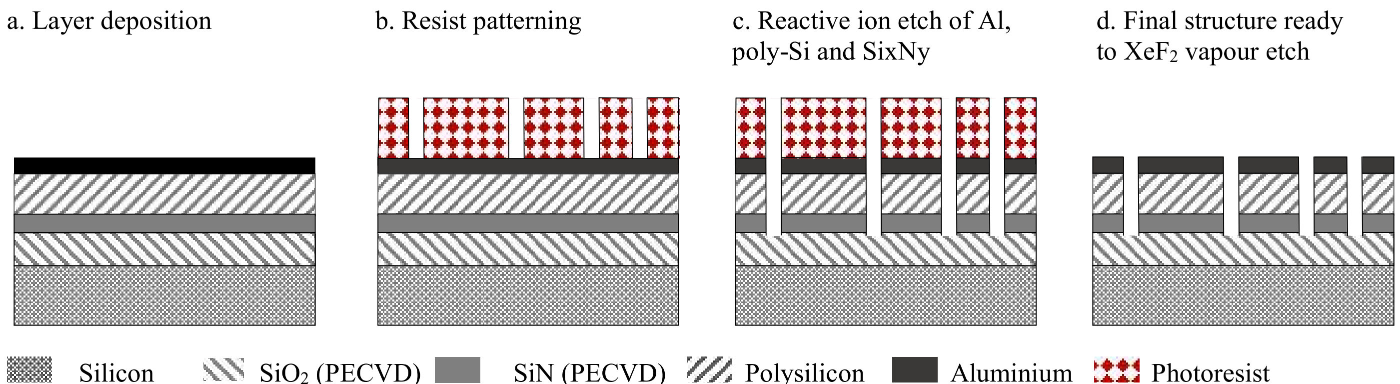

Fig. 3. Schematic process flow of the fabrication of a XeF_2 vapour etch selectivity test structure

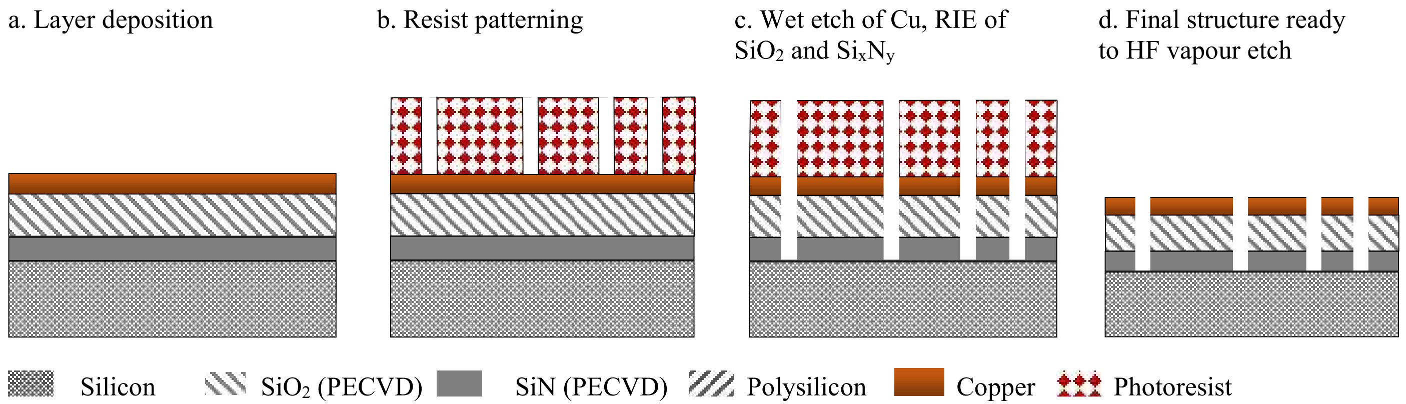

Fig. 4. Schematic process flow of the fabrication of a HF vapour etch selectivity test structure

widest bridge that is displaced by the height of both the sacrificial and the structural layers. Figure 1 shows a schematic cross-section of one of the bridges being gradually released and the stylus displacing the bridge during the measurement.

III. Test Structure Design

A. Layout

The test structure is formed of a stack of deposited thin films, comprising a structural layer, a sacrificial layer and finally a capping layer. The XeF_2 structure reported here uses a PECVD silicon nitride structural layer in combination with a LPCVD polysilicon sacrificial layer. These are capped by a layer of aluminium that is unaffected by the XeF_2 vapour etch. The HF test structure also uses a PECVD silicon nitride structural layer. However, in this case, a PECVD silicon dioxide sacrificial layer and a copper capping layer is used.

In both cases, trenches are etched in the layer stack using a reactive ion etch, which defines the bridge structures and exposes the layer edges to the vapour etch process. As part of the design the bridge width is incrementally reduced and the layout is shown in figure 2. For measurements of large undercuts the bridge width decreases from 100 to 52 µm in increments of 2 µm. For smaller undercuts, narrower bridges are defined with widths from 50 to 2 µm in increments of 1 µm.

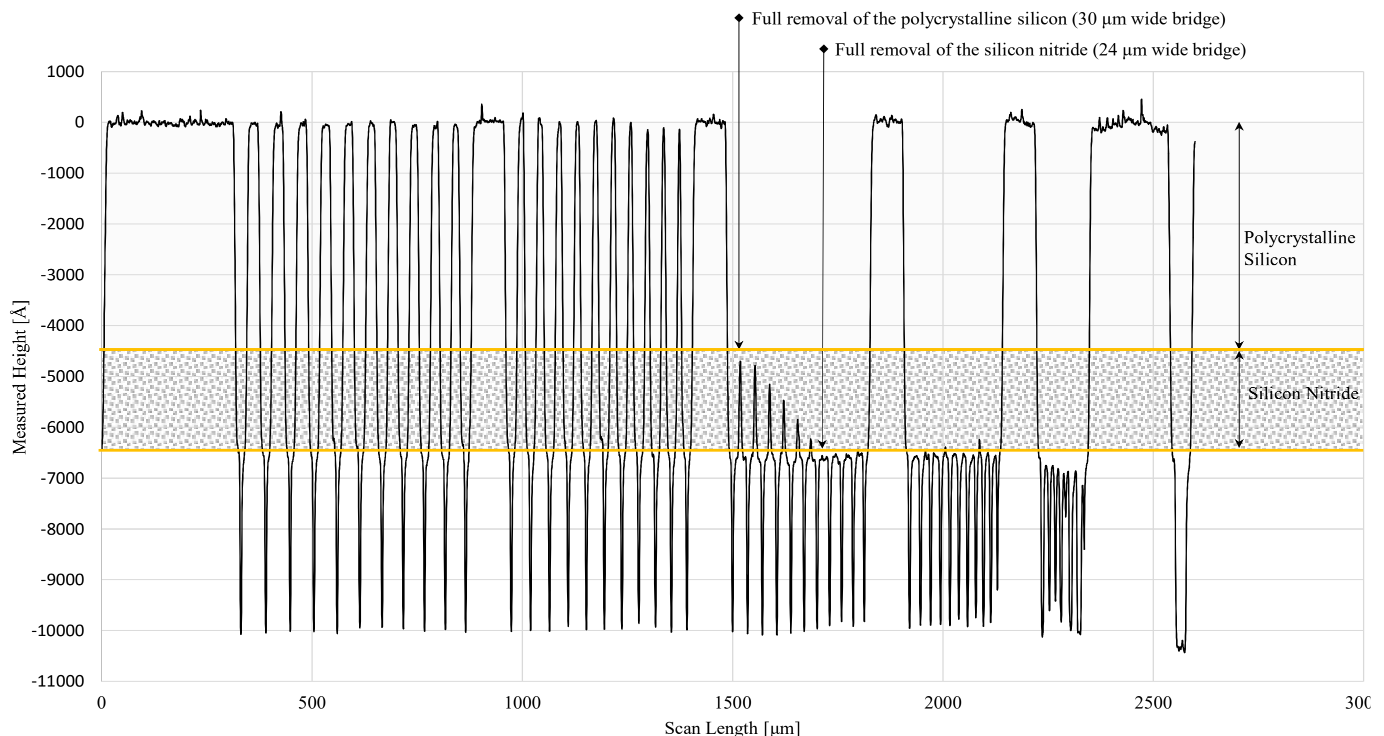

Fig. 5. Surface profile retrieved after vapour etching the XeF_2 test structure. The thicknesses of the polycrystalline silicon and silicon nitride layers are represented by the different underlyings of the graph.

The bridge structure has a number of advantages over alternative architectures. It is less prone to stiction issues during scanning, enabling a robust extraction of the 1D etch front propagation. In addition, it gives less noisy deflection measurements compared with other designs such as the cantilever structures of [3]. Finally each scan should have the format shown in figure 5 thereby confirming the correct operation of the test structure.

For the initial investigation, bridges with lengths of 100 µm, 200 µm and 300 µm were employed. The measured etch undercuts and selectivities were found to be independent of the bridge length. However, the alignment of the shorter test structures for the automated profilometer measurement of multiple test structures becomes very time-consuming. A bridge length of 300 µm was selected as it requires minimal wafer real estate for the test structure, while the layer thicknesses between 200-500 nm can withstand the vertical and horizontal displacement during measurement. The 5 µm trench width between bridges enables the tip of the profilometer stylus to measure the full depth of the trenches and simplifies the photolithography.

B. Layer Configuration

The test structure can be used to measure the selectivity of various materials in close proximity. For example, figure 3 shows the fabrication process flow for a test structure used to determine the etch selectivity of polycrystalline silicon versus silicon nitride in XeF_2 vapour etching. The structural layer is placed below the sacrificial layer, which allows the etch rates of the two materials to be determined with a single surface profilometry measurement.

In this example, the 500 nm thick silicon dioxide etch stop layer and the 210 nm thick silicon nitride structural layer were deposited using plasma enhanced chemical vapour deposition (PECVD) at low and high frequencies respectively. Silicon dioxide was selected as an etch stop because it is largely unaffected by the XeF_2 vapour etch process while remaining stable at high temperatures. If the etchant used attacks silicon dioxide, a 50 nm thick platinum layer can be used as an alternative etch stop. The 450 nm thick sacrificial layer of polycrystalline silicon and 350 nm thick capping layer of aluminum were deposited using LPCVD and sputter deposition respectively.

Aluminum was employed as the capping layer, because it is not attacked by the XeF_2 vapour process, can be sputter deposited and reactive ion etched, while its mechanical properties prevent fracture of the bridges during the profilometer measurement. Aluminum is not recommended as the first choice capping layer for hydrogen fluoride vapour etching, because it can fluorinate and the presence of the resulting particulates on the sample creates noise during the measurement. Instead, we suggest using copper as the capping layer for the hydrogen fluoride test structures. It is resistant to HF, does not fluorinate, and in contrast to polysilicon, the residual stress within the layers can be controlled by straightforward annealing processes. The stress control method will be briefly elaborated on in a later section of this work.

The remaining architecture of the HF test structure is very similar to the one for XeF_2 and is displayed in figure 4. A 500 nm thick PECVD silicon dioxide was used as a sacrificial layer along with a 250 nm PECVD silicon nitride structural layer. An etch stop layer is not required for the HF test structure because hydrogen fluoride does not attack the underlying silicon wafer.

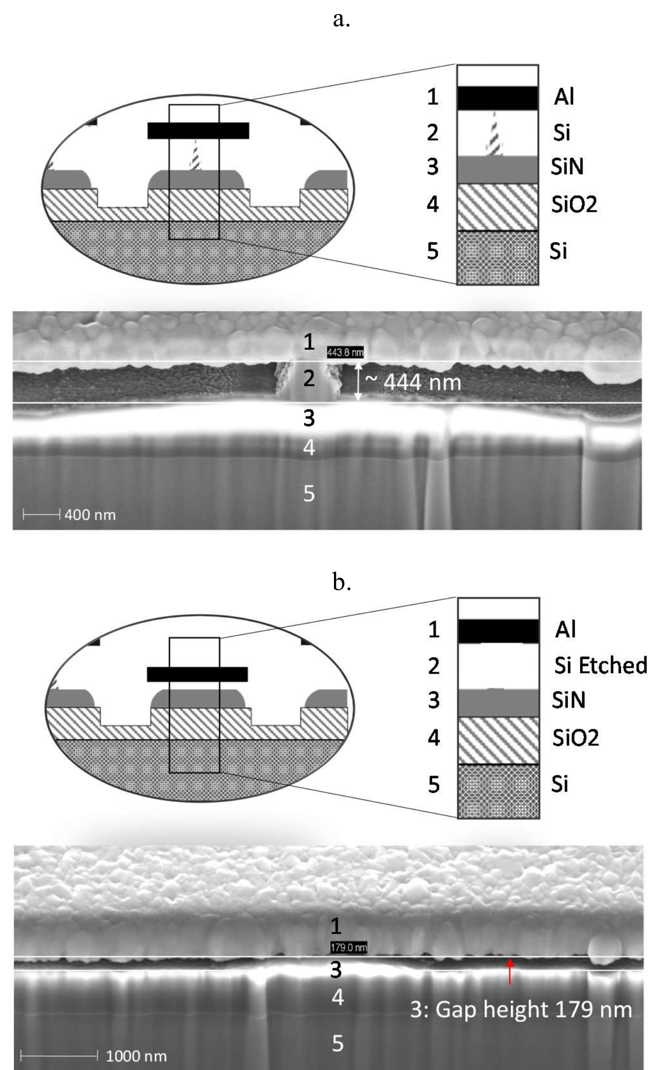

Fig. 6. Scattering electron microscope (SEM) images of focus ion beam (FIB) cut crossesctions of a. the centre of the 31 µm wide bridge. The polycrystalline silicon pillar has not been fully removed yet and prevents deflection of the bridge during profiling. b. The polycrystalline silicon layer of the 28 µm wide bridge has been fully etched. The bridge has deflected and is supported by the remaining silicon nitride.

The patterning process is the same for both structures. A contact mask aligner was used to transfer the pattern from a chromium photomask into a 3 µm thick layer of photoresist (SPR 220 – 3.0) which was subsequently developed for 1 minute in MF-26. In the case of the XeF_2 test structures, the trenches in the aluminum, polycrystalline silicon, and silicon nitride layer stack were reactive ion etched. In the case of the HF test structures, the copper was wet etched, and the silicon dioxide and silicon nitride were reactive ion etched. After the resist was removed, the wafer was diced into 90 chips with dimensions of 11 x 5.5 mm.

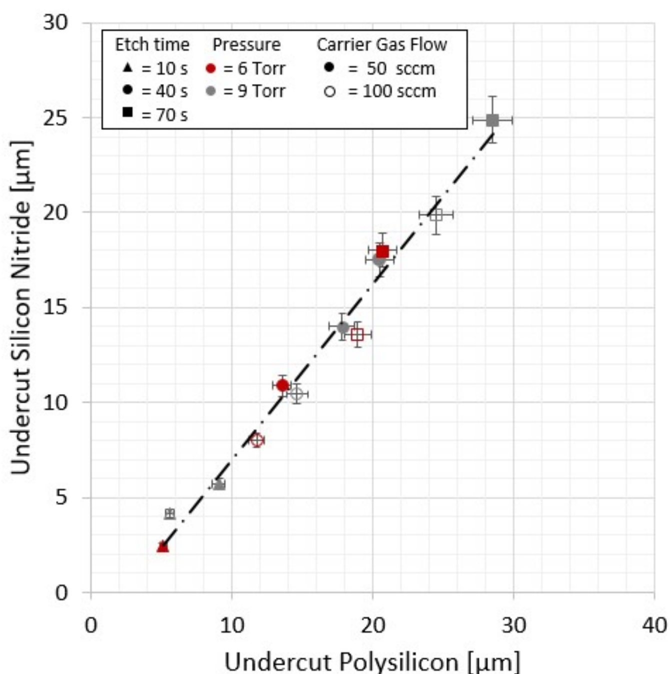

Fig. 7 This example dataset presents the selectivity between polysilicon and PECVD silicon nitride. It is based on 12 samples with the layer configuration and design presented in section III. The etch parametes can be derived from the indivdual markers design. The etchant was gassous XeF_2.

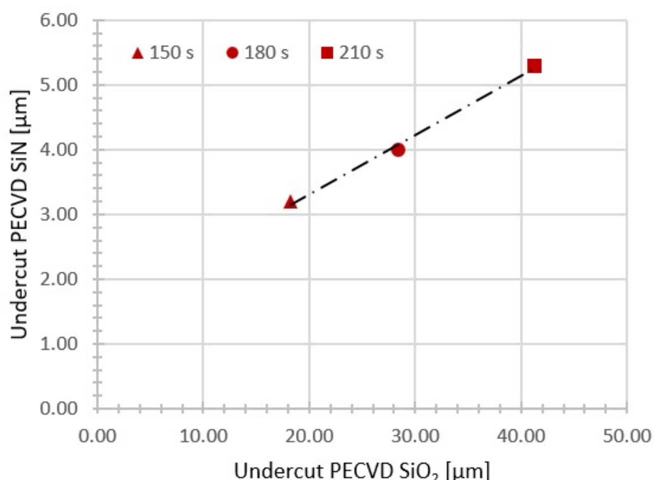

Fig. 8 This example dataset presents the selectivity of PECVD SiO_2 over PECVD SiN. It is based on three samples, that were etch at a pressure of 11 Torr at a temperature of 20°C. The HF, H_2O and N_2 flows were 61, 95 and 9 sccm respectivley. The etch time can be derived from the shape of the markers.

IV. INTERPRETING THE SURFACE PROFILE

After the test structures have been scanned by the profilometer the resulting surface profile is used to obtain the undercut for the sacrificial and the structural layer. The measurement reading procedure is explained on the basis of test structures that were exposed to a xenon difluoride vapour for 40 seconds at a process pressure of 9 Torr, with a nitrogen carrier gas flow of 100 sccm, at a temperature of 30°C.

The resulting surface profile is displayed in figure 5. For these particular process parameters, the 30 µm wide bridge was the widest one that has been vertically deflected by more than 450 nm (the thickness of the polysilicon) indicating the sacrificial material has been fully etched. The resulting undercut is 15 µm with an apparent etch rate of 375 nm s^{-1}. The 24 µm wide bridge has deflected by 650 nm (the combined thickness of the polysilicon and silicon nitride layers), indicating the structural layer of silicon nitride has also been fully etched with an apparent etch rate of 300 nm s^{-1}. This suggests an etch selectivity between the polysilicon and silicon nitride layers of 5:4. Depending on the output format of the profilometer used, programming can be used to automatically extract the undercut data from the surface profile.

V. MEASUREMENT VERFICIATION

The test structure measurements were verified using two different methods for the XeF_2 etched samples. As the physical mechanisms underlying both the selectivity measurement and verification methods are the same in each case, it was not necessary to repeat this for the HF test structures. Firstly, five samples from a larger pool of chips, that were XeF_2 etched and measured during the process calibration, were randomly selected and the capping aluminum layer removed. Energy-dispersive X-Ray spectroscopy (EDX) was used to determine if the silicon nitride was removed at the bridge width indicated by the surface profilometry. In all cases, the EDX measurement agreed with the results of the surface profile. Secondly, cross sectional SEM images of etched structures confirmed that the polycrystalline silicon and silicon nitride were removed under the bridge, indicated by the test structure.

Figure 6a shows the 31 µm wide bridge being supported by a thin pillar of sacrificial polysilicon. Figure 6b shows the 28 µm wide bridge of the same test structure. In this case the sacrificial layer has been fully removed and the aluminum bridge is suspended above the silicon nitride layer.

VI. RESULT AND DISCUSSION

A. Experimental Result

A series of etch experiments was undertaken on a XeF_2 vapour etch tool, to characterize the performance of the test structures. The tool operates in a continuous flow configuration, constantly supplying the xenon difluoride to the etch chamber. It has the capability to control the processing pressure, chamber temperature and enabls additional supply gasses to be introduced into the etch chamber.

The nitrogen carrier gas flows through a bubbler, introducing the xenon difluoride into the chamber. The xenon difluoride concentration within the etch chamber is inversed proportional to the carrier gas flow. An excerpt of a larger dataset is displayed in figure 7, with each of the 12 data points representing a sample. They were etched at different process pressures, carrier gas flows and etch times at a constant temperature of 25°C. Eight test structures were measured on each sample. The average measurement and the etch parameters used are presented in figure 7 and the error bars indicate the standard deviation of each

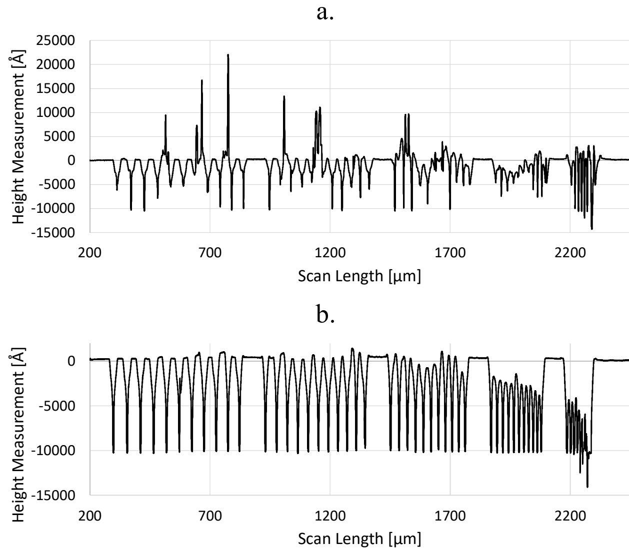

Fig. 9 A comparison of the HF test structure's post etch surface profiles. In (a) without heat treatment, and in (b) after annealing the etched test structure on a hotplate, set to 170°C for 60 seconds.

dataset. The data is consistent and shows a polycrystalline silicon to silicon nitride selectivity of 5:4. Furthermore, it shows that the selectivity is independent of the processing pressure, xenon difluoride concentration and etch time. The reasons for this and methods to significantly improve the selectivity have been recently reported by the authors [15] .

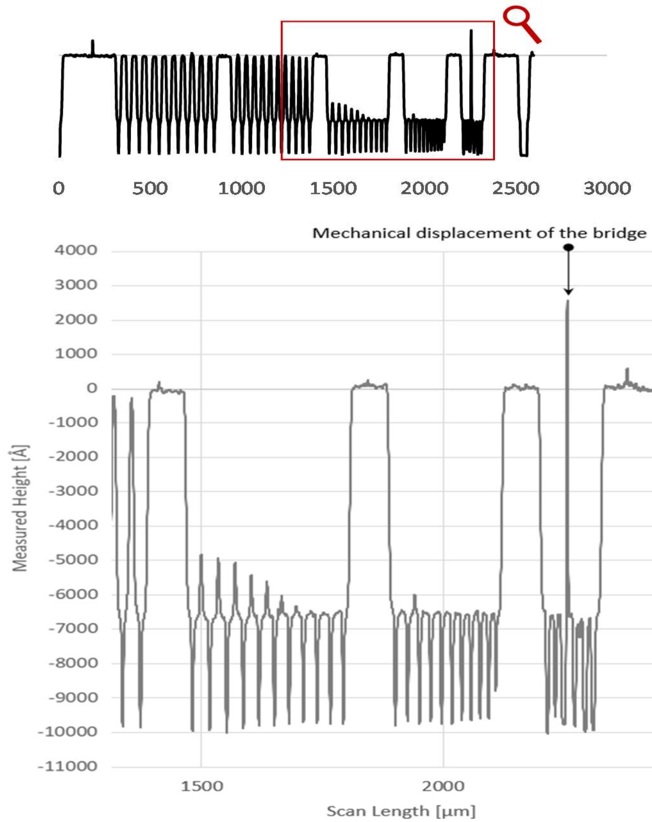

Fig 10. This surface profile was obtained from the same sample as figure 5's. The signal is imprecise due to a horizontal displacement of a 8 µm wide bridge caused by a too high scan speed.

In addition to this, some preliminary data for an HF etch selectivity study is presented in figure 8. The dataset is less extensive than the one presented for XeF_2 processing, because in order to maintain comparable experimental etch conditions over a broader temperature and concentration range the process must operate at the vapour pressure of the gas mixture. Hence, there is a single set of pressure, HF and H_2O flows for each set of processing temperature and HF gas concentration parameters.

The data presented was obtained at a processing temperature of 20°C at an HF gas concentration of 37%. However, it is important to take into account the processing time, because the memsstar Xeric HF etch system used in this study requires up to 40 seconds to stabilise the gas flows. The dataset displayed in figure 8 suggests that the selectivity of PECVD silicon dioxide over PECVD silicon nitride increases over time from roughly 6:1 at an etch time of 150 seconds to 8:1 at an etch time of 210 seconds. The reasons for this and methods to improve the HF etch selectivity are currently being investigated by the authors.

B. *Performance*

A total of 56 chips with 8 test structures per chip, were vapour etched in a XeF_2 atmosphere at different conditions to characterize the resulting variance in the etch undercuts of the structures on each sample. The maximum polysilicon and silicon nitride undercut standard deviations were 1.96 µm (mean 27.86 µm) and 2.2 µm (mean 28.5 µm) respectively. The population standard deviations and the yield of successful measurements are displayed in table 1. The performance can be significantly improved by adopting the design and measurement considerations presented in the next section.

TABLE I.
PERFORMANCE OF XeF_2 TESTS STRUCTURE

	Polysilicon	SiN
Number of attempted measurements	448	448
Number of successful measurements	426	350
Successful measurements as %	95	75
Population Standard Deviation σ [µm]	0.4	0.37

C. *Test Structure Measurement Consideration*

The authors observed six modes that can cause a faulty or incomplete measurement. Firstly, it is important to carefully define the etch time to prevent both over and under etching. In this example, over-etching would occur once the 100 µm wide (widest) bridge is released, because in that case the maximum undercut cannot be determined. Similarly, no measurement can be obtained, if twice the undercut is less than the width of the narrowest bridge. Clearly, the test structure layout and the process to be evaluated are important considerations during the design process.

Residual stress of the capping layer can result in faulty measurements because the bridges buckle and can not be displaced by the profiler stylus. Such a profile is displayed in figure 9a. This issue was observed for the copper capping layers during the development of the HF test structure, while the aluminium capping layer of the XeF_2 test structure was not affected. Post vapour etch annealing of the HF test structures was experimentally investigated to resolve this issue.

As suggested by the literature [19], a strong response of the residual stress was observed for the temperature range of 160 – 210 °C. The optimum process, an example is displayed in figure 9b, was achieved when heating the sample to 170 °C for the duration of 60 seconds on a hotplate. In this process, the stress within the layer moves from tensile to compressive. Should the stress in the copper be non-optimal, then another successful approach is to scan the bridge array twice, first with a low downforce (< 3mg) to obtain the deflection of the narrow bridges and a second time with a large downforce (> 10 mg) to deflect the larger bridges.

The three remaining measurement issues are caused by sub-optimal surface profiler settings. Firstly, mechanical destruction of the bridges can occur if the downforce of the surface profiler's stylus is too high. In consequence, the bridges are ripped from their anchoring, adhere to the stylus and contaminate the tool. Secondly, the stylus bounces off the bridges if the downward force is too low. This leads to a perturbed signal that resembles a positive bridge deflection up to 10 micrometres. A downforce of 2 – 3 mg yielded the best results for the samples presented in this study.

The measurement conditions identified in figure 10 result from a mechanical deflection of a narrow bridge caused by too high a scan speed. This has only been observed on bridges that were narrower than 10 µm at scan speeds higher than 40 $\mu m\ s^{-1}$. Hence robust measurements can be achived by reducing the scan speeds for samples were the accurate measurement of this segment of the test structure is essential.

VII. Conclusions

This paper and [16] have reported for the first time a bridge based test structure that can characterize the isotropic etch selectivity between two materials under realistic MEMS fabrication conditions. The test structure is designed to be used in vapour etch processes but can also be adapted for wet etch release processes. It can be employed to characterize a wide range of materials with the fabrication of the test structure being quick and straightforward.

The measurement methodology has been demonstrated by HF and XeF_2 etching of example stacks of layer materials, which may not be those that would be used in a commercial process. However, they provide an experimental dataset that clearly shows that these test structures deliver coherent measurement information. The design can be employed in industrial MEMS fabrication processes, with the area required being small enough to be placed on production wafers.

The test structure can be easily adapted to accommodate different dimensional requirements. The measurements taken with this test structure are robust, because faulty measurements resulting from broken or contaminated bridges are evident from the profiler signal. Individual test structure arrays can be manually measured within 15 seconds. Large numbers of devices can be assessed by automating the process. For instance, an automatic measurement algorithm was used on the Bruker Dektak XT profilometer to measure the test structures reported in this study. Typically this can measure about 200 test structures within 2 hours.

Acknowlegement

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Datashare

The data presented in this work is available online at https://doi.org/10.7488/ds/2962 .

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