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Membrane platforms for sensors

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Abstract

In the past half century the IC technology could produce an unprecedented growth and penetration into all segments of our daily life due to the achieved extreme productivity and reliability by using well established and continuously refined processing platforms. This uninterrupted trend continued also with the advent of the More than Moore-type MEMS technology allowing the combination of the signal processing capability of the mature IC technology with the exploitation of mechanical, thermal, optical properties of the materials used in IC technology in different sensing and actuation purposes. Mass producibility by monolithic integration required here also the development of appropriate unified set of techniques for the various applications, i.e. sort of standardised platforms to explore.
In this paper we focus on the development of bulk micromachined membrane platforms, being exploited in such applications. At the same time this summary also offers a "case study", a retrospective review of the development of integrable sensors from pressure measurement to nanopore-type, label-free biosensing at the Institute of Technical Physics & Materials Science - MFA, Budapest.

Keywords: MEMS, membrane, bulk micromachining

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1. Introduction

Already in the early nineties two distinct techniques emerged for the integration by silicon MEMS, which are

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known by the community as *surface micromachining* and *bulk micromachining*. An early review of the field by P.J. French and P.M. Sarro surveyed the field and the related requirements [1]. They stated that even if starting from the ‘80s the polycrystalline silicon based surface micromachining became most widespread, bulk micromachining was the first to utilise mechanical properties of thin membranes e.g. as diaphragms in pressure sensors [2]. The parallel route of surface and bulk micromachining led to a “specialisation” which manifests itself in different process complexity and dimensions (up to two orders of magnitude smaller in surface micromachining). There are, however, heavy duty niche applications, where the robustness of bulk micromachining is needed in both, active and passive applications. They cannot be fulfilled by the cheaper and by now more mature surface micromachined solutions.

Usually, the term membrane is connected to solutions for the separation or filtering of gaseous or liquid mass flows using e.g. permeable membrane approaches. *Thin suspended films*, however, are not only applied in such context, but also as mechanical support in general, or substrate in the realisation of microsensors in the physical, chemical or biochemical domain. In such applications the thermo- and electromechanical, but often also optical properties are rather made use of. The inorganic or organic thin supporting membranes can be insulating or semiconducting, depending on the applications, in form of a contiguous (full membrane) or patterned (perforated membrane) film. Design and processing constraints of these delicate structures, however, often limit the functionality, stability and reliability of the MEMS device, which is composed using these elements.

Below the capabilities, influencing factors and characterisation of these membrane platforms will be discussed based on practical examples from the R&D results of MFA in different physical, chemical and bio-sensor applications.

2. Overview of membrane processing and applications at MFA

2.1. Membranes by alkaline etching

Pressure sensors were the first mass product making use of the favourable mechanical properties of single crystal silicon in exploration of the excellent piezoresistive response in a wide pressure range. As the piezoresistors are placed on the front side of the (100) oriented silicon substrate, a double-side alignment is needed for recessing the backside appropriately. The formation of the membrane was achieved first by mechanical thinning, giving rise to severe misalignment problems. Later alkaline wet chemical etching was introduced [3] using EDP (Ethylene Diamine Pyrochatechol), TMAH (Tetramethylammonium Hydroxide) or KOH with much better accuracy but requiring a/o. etch stop techniques and the suppression of enhanced etch-rates along the perimeter of the diaphragm, especially for thin (5-20µm) membrane formation. The well controlled thickness and uniformity led to the production of thin membranes and facilitated the introduction of capacitive type pressure sensors using wafer bonding as well. (Fig.1).

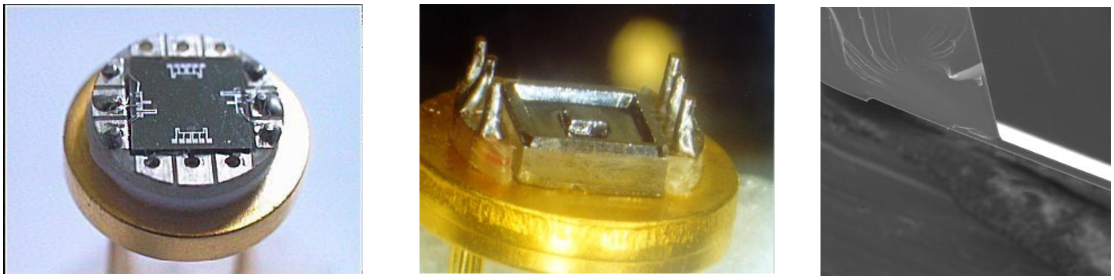

Fig. 1. Pressure sensors formed by preferential alkaline etching. Piezoresistive -(left) and capacitive-type (centre) sensor chips mounted. The SEM cross-section of a cleaved Si element of the capacitive sensor element reflects the excellent uniformity of the 10µm thick membrane (right).

This technology could also be adapted to the formation of photo-acoustic pressure sensor by integrating two cantilever type mirrors over the active and reference photoacoustic channels. The principle of read-out here is the measurement of membrane displacement via the reflected focused laser beam. These early experiences laid the fundament to more sophisticated membrane platforms to be elaborated.

2.2. Membranes by porous silicon sacrificial layers

The way to avoid the need for double side alignment in silicon processing was opened by the introduction of anodic etching of single crystal silicon, i.e. by the formation of porous Si sacrificial layers in bulk micromachining [4]. Although well controllable, this technique did not easily allow the fabrication of "full membranes". It produced, however, after the selective removal of the sacrificial porous layer suspended single crystal structures, a kind of "perforated type" membrane in silicon purely from the front side. Moreover, the technique may provide an attractive combination of bulk and surface micromachining features in terms of membrane and cavity geometry. The porous layer protruded namely sideway under the structure to be suspended, and in the self-stopping anodisation process the etching fronts from both sides even came so close to each-other that a subsequent wet-chemical etch could completely release the suspended structure. The membrane could be formed entirely from n-type single-crystalline silicon or from a combination of silicon and Si rich silicon-nitride, too. Due to the HF-based electrolyte required for the electrochemical process, however, Si_3N_4 and SiO_2 cannot be considered as structural material. The advantages were obvious: not only the thermal isolation from the silicon bulk could be realised, but - albeit with certain restrictions - also the advantage of the manufacturing of electronic devices on the suspended Si was maintained.

The suspended, Pt-connected bridges could be used as thermally isolated "heater filaments", temperatures up to >1000 C° were achieved at reduced power dissipation as low as 0.025mW/^{o}C (Fig.2) with stable device operation up to 550^{o}C. Above his temperature the degradation of the metal contacts on the low heat-capacity Si filaments rapidly deteriorates the structure.

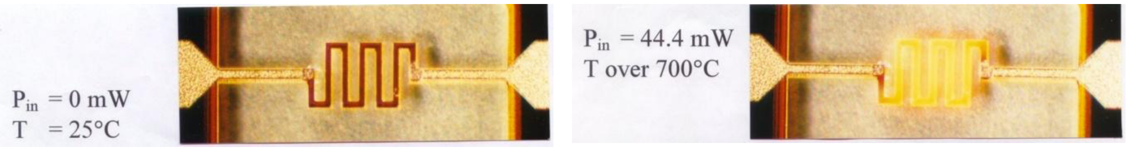

Fig. 2. Single crystalline Si micro filament suspended by Pt-wiring over a deep (>20µm) cavity formed by porous Si micromachining. The right image shows the 5 µm wide filament of reduced power dissipation glowing.

Some groups tried to achieve a similar performance with an embedded heater and left the heat-isolating porous layer in place leading to better mechanical stability [5]. In our case the fragility of the entirely suspended beam, or heater element could be reduced by leaving a thin support (like a fin) underneath the heater element after the well controlled removal of the sacrificial porous layer (see in Fig. 3). Due to the depletion of carriers inside this supporting single-crystalline pillar (causing the "self-stopping" of anodisation), the heat conduction in this structure remained comparable to the level achieved by air gap isolation after complete removal of sacrificial porous Si.

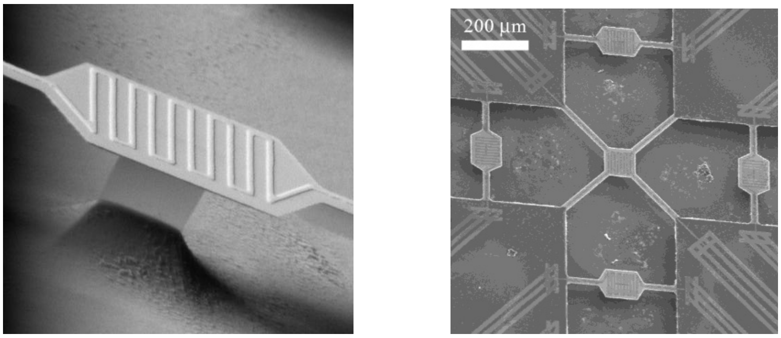

Fig. 3. Suspended microheater stabilized with a thin Si support (left). Direction dependent flow sensor constructed with a heater element in the centre surrounded by four temperature reading resistors suspended in the channels (right).

The process offered itself for the realisation of reliable heater elements to be used e.g. in *gas and flow sensing at elevated temperature, or as fast IR source* in various applications. Soon it turned out that the heat isolation to be used in combustion-type gas sensing does not require the heater to be made of single crystal silicon. Instead, doped poly-Si or Pt heaters on a thin insulating membrane can do the job better. This is definitely true for devices utilizing temperature read-out of filaments in the calorimetric-type gas sensing [6] (Fig.3)

At this point our attention was rather focused to the exploitation of the *piezoresistive properties in the suspended single crystal by porous silicon micromachining*. Since the read-out of piezoresistors in Wheatstone-bridge arrangement requires the addressing and on-chip amplification of the signal, the CMOS-compatibility of the established *perforated membrane platform* became a must for monolithic integration. By keeping the processing temperatures required for the 3D structure fabrication below the Al sintering temperature, the sequence for bulk micromachining can be implemented after the fabrication of the circuitry (preferably with a foundry-compatible process) was completed [7]. This patented solution allowed the start of the very successful development of 3D force sensors and their integration to a tactile sensor array – still by using the front-side porous Si micromachining (Fig.4).

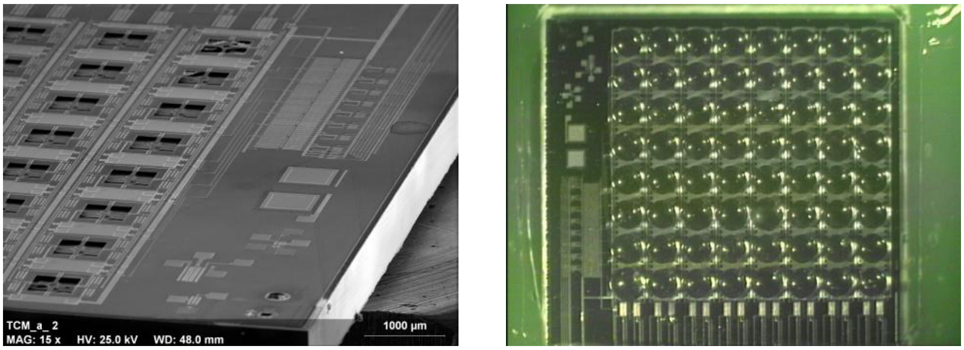

Fig. 4. The “electronic fingertip”, a 64 element 3D micro force sensor array using the piezoresistive principle. Each 400x400 μm^2 element (taxel) is a vectorial force sensor in the tactile array, which senses the components of the attacking force vector focussed onto the suspended cross-bridge with the four piezoresistors in the arms. CMOS compatibility of porous Si micromachining is demonstrated by the use of the integrated multiplexer (left). The encapsulated tactile sensor chip is protected by the elastic silicone rubber with hemi-spherical “fingerprints” performing the focussing of the mechanical force (right).

2.3. Membranes released by deep reactive ion etching - the thermo-mechanical issue

Membranes in sensors are often built from multilayers and carry patterned materials to serve the function the device is designed for. Thereby the complex structure is constructed from materials of different thermal and mechanical properties, and is exposed to thermal shocks during fabrication and sometimes during device operation as well. This is manifested in the emerging mechanical stress and deformation of the membrane.

The microheater-based gas sensor development took a different path by not any more requiring the frontside micromachining. The heat isolating membranes holding the embedded heater elements for these *harsh applications* could better be formed by stress-free isolating multilayer stacks contrary to the porous Si micromachining alternative. Although reduced stress Si-rich nitride layer is also compatible, the lower heat conductivity of stacks of silicon dioxide and nitride can further reduce the power needed for heating. In order to fulfill the minimum power requirement, sensor devices operated at elevated temperature must exhibit perfect thermal isolation. A reliable dynamic operation under such circumstances constitutes an even more severe requirement. Consequently, the most critical issue is a proper thermo-mechanical design. For the optimisation Finite Element Modeling by the COMSOL Multiphysics code was used as demonstrated for the case of the embedded microheater in Fig. 5 [8].

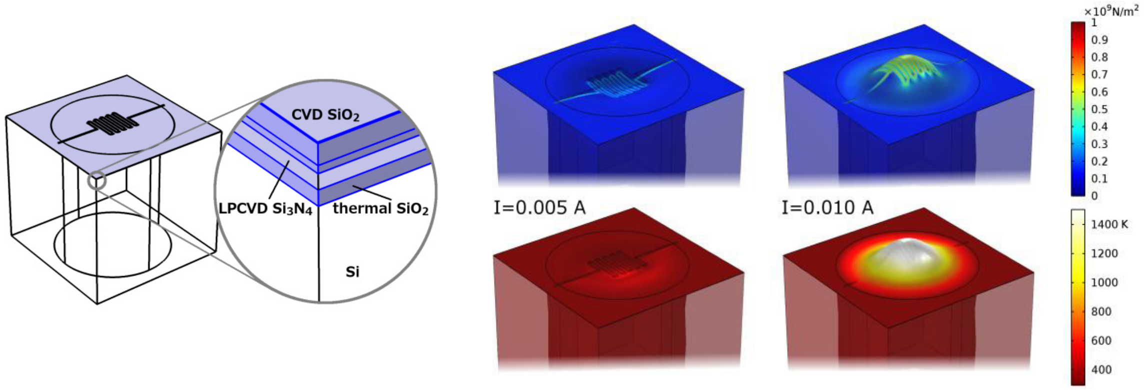

Fig. 5. Membrane stacking in a 400 □m×400 □m microheater model (left). Distribution of Von Mises stress (top) and temperature (bottom) calculated by COMSOL Multiphysics for the simulated micro-heater driven by 5 mA (left) and 10 mA (right). Note, the vertical displacement is shown exaggerated 50× (right) [8].

The *combustion-type gas sensing of explosives at a minimum power consumption* was for long an unsolved problem, as the power requirements are very much restricted by the international transducer norms. Using the MFA membrane platform for the manufacturing of "*micropellistors*" we succeeded in offering a scheme for fulfilling both the transducer norms and the required functionality [8].

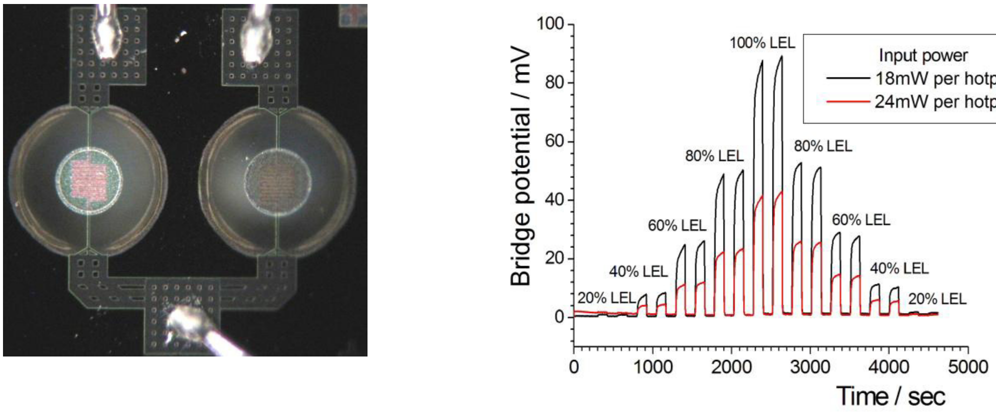

Fig. 6. The *micropellistor* consists of two hotplates of identical thermal properties on full multi-stack isolating membranes of 300 µm diameter. The right one in the picture is coated with catalyst (Pt) supported by porous Alox, the other serves as passive reference (left). Pellistor response on propane at different operational temperatures (right).

The excellent heat isolation of large area, stress-free membrane structure was also exploited in the formation of integrated thermopile arrays (Fig. 7) as designed for IR detection or radiation in the mm wavelength range [9]. The bulk micromachined chip contained a large size insulating stress-free multilayer membrane stack of 3300x900µm^2.

Characterisation of the geometry and the thermo-mechanical properties of the membrane elements, and continuous feedback into design and technology is necessary at all stages of the research and product development. Makyoh topography is an optical method for flatness and defect characterisation of nearly flat mirror-like surfaces. The concave (or convex) irregularities locally focus (or defocus) the reflected light, causing contrast variations in the reflected image (see in Fig. 7). The principle using a homogenous collimated light source and defocused detection system with additional optics and CCD cameras was adopted at MFA for the qualitative inspection of the semiconductor wafer surface, the basic imaging parameter is the screen-to-sample distance [10]. The method can be made quantitative for the extraction of stress parameters by inserting a structured mask, e.g. a square grid into the path of the illuminating beam, the smallest measured membrane centre deflection was about 50 nm. The smallest membrane size that could be studied was 4 mm [11].

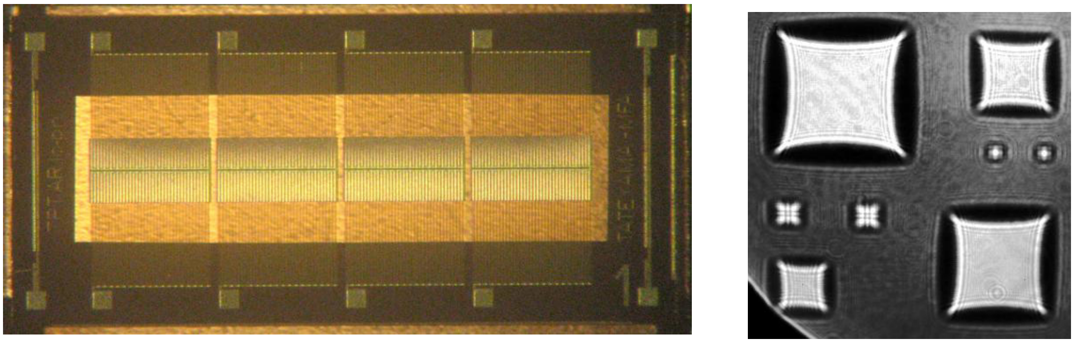

Fig. 7. Four element thermopile array formed on a single large size membrane of 3300x900μm^2 [ ](left). Full wafer Makyoh-topography image of SiN/Si membrane structures. The square membrane side lengths are: 1 mm, 2 mm, 4 mm, 6 mm, 8 mm and 10 mm, respectively. The pillow shape of the membranes' images indicates increasing inclination of the edge region towards the side centre point, i.e. the membranes have a dome-like shape. The smallest detected centre deflection assuming a spherical dome shape from the comparison to interferometry was 700nm in these measurements (right).

2.4. Solid-state nanopore menbranes for biochemical sensing

As mentioned in the introduction the original conventional application of porous membranes is the separation or filtering of targeted species from liquid samples. The challenging biomedical task of cell and molecule recognition or counting opens up a novel application field for special membrane platforms in MEMS/NEMS devices. Membrane platforms with chemically modified nanonopores are reported to facilitate extreme high sensitivity detection of specific biomolecules (marker proteins, viruses, RNA). They recognise the targeted species in a label-free manner by binding in or translocating through the pores, thereby significantly changing the pore impedance [12, 13]. This transport modulation through the nanopore is envisaged as fundamental operation principle for a new generation, *nanoelectronics-based high sensitivity, label-free medical diagnostic or DNA sequencing platform*. They may provide the obvious solutions in the development of diagnostic Lab-on-a-Chip systems e.g. for the diagnosis of cardiovascular diseases (CVD) through relevant biomarkers. The reliability and reproducibility of nanoscale fabrication processes, however, remains a challenge for manufacturing.

Biosensor application with single molecule sensitivity using this sensing principle poses extremely strict chemical, electrical and mechanical requirements for the nanoscale technology. Precisely tailored and reproducible nanopore array formation by the combination of silicon bulk micromachining and subsequent nanofabrication steps had to be established for different membrane structures. To ensure the proper chemical and electrical resistance as well as low residual stress of the stacked SiO_2/SiN_x supporting membrane the appropriate composition with adequate layer thickness ratio is extremely critical. Moreover, also *the geometric parameters of the pores had to be tuned according to the requirements of the different targeted biomolecules and pore-surface functionalisation methods. A typical multilayer membrane structure with a pore array is presented in Fig. 8.* Focused (Ga^+) Ion Beam (FIB) milling of nanopores in silicon-nitride and gold coated silicon-nitride layers provided the controlled pore geometries in the mechanically stable membranes for bio-functionalisation. These were integrated into an electrochemically addressable fluidic system comprising the fluidic channels and electrodes (Fig. 8).

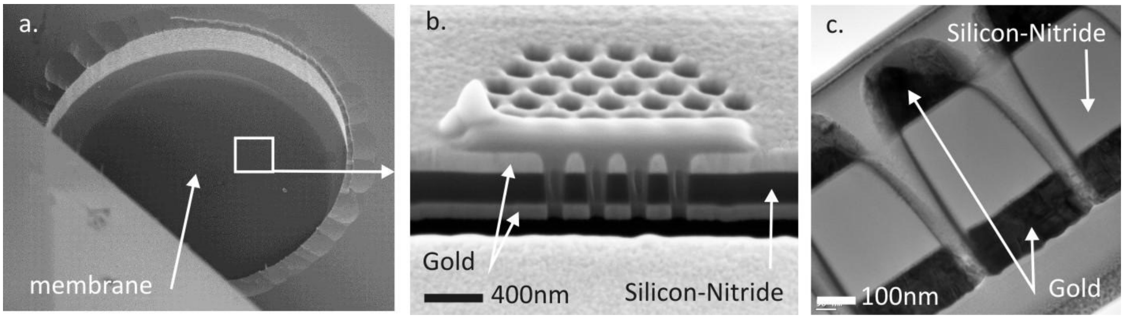

Fig. 8. SEM image of the microfluidically integrated, deep reactive ion-etched (DRIE) membrane (a.). Six of such nanopore chips for multi-parallel measurements were mounted into a biosensor cartridge. Cross-sectional view of the structure of the Focused Ion Beam milled nanopore array on a SEM (b.) and TEM (c.) microimage.

3. Conclusions

Although the scene in MEMS manufacturing today is dominated by surface micromachining, bulk micromachining is having a fair share in „heavy duty" niche applications. Materials and device design both are essential for facilitating the special device performance, which makes bulk micromachined membrane-type sensors an ideal research topic for small laboratories as well. By maintaining the IC compatibility even mass products can be developed in simple fabless circumstances, provided the key issues of thermo-mechanical stability can be properly managed.

4. Acknowledgement

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