Ultrathin TiN Membranes as a Technology Platform for CMOS-Integrated MEMS and BioMEMS Devices


A standard complementary metal-oxide-semiconductor (CMOS) process is successfully modified to encompass the preparation of suspended TiN membranes of only 50 nm thickness from one of the metal layer stacks of the back-end flow. The layers’ elastomechanical constants are determined with high precision by laser Doppler vibrometry. Residual stress gradients are compensated and a state of moderate tensile strain is introduced into the membranes. Test systems of TiN beams and bridges operating in a capacitive coupling scheme are optimized for the low voltage range attainable with CMOS devices. TiN actuators are particularly suited for applications in biotechnology like sensing of pressure or viscosity in microfluidic devices due to their high corrosion resistance in liquid electrolyte surroundings. The established inclusion of the process in a CMOS pilot line enables the production of cheap and monolithically integrated microelectromechanical systems (MEMS) and bio-microelectromechanical systems (BioMEMS) devices.

1. Introduction

The on-going scaling in microelectronics following Moore’s law is increasingly supplemented by including additional functionalities in a “more than Moore” approach. Prominent examples are given by microelectromechanical systems (MEMS) that became widely used for sensing physical quantities like acceleration, force, torque, pressure, etc., and which might also apply to so-called BioMEMS for sensing biomedical parameters or interacting with biomolecules. The elastic components of MEMS devices are usually prepared from single- or polycrystalline silicon layers, thereby restricting mechanical properties to those of silicon. It would be desirable to extend the spectrum of measurable physicochemical properties, sensitivities, and materials in particular, BioMEMS have to withstand severe corrosion in physiological milieus. Here, we report on ultrathin TiN membranes from the back-end-of-line stack and the precise determination of their elastomechanical constants by laser Doppler vibrometry. The availability of such layers from a standard complementary metal-oxide-semiconductor (CMOS) flow enables new and inherently integrated MEMS and BioMEMS devices.

Active structures in MEMS devices often exhibit the shape of suspended beams with lateral dimensions of some ten to hundreds of μm, while their vertical extensions remain smaller. Particularly in electrostatic MEMS actuation schemes the beam thickness may enter the sub-μm range. Therefore, in addition to silicon layers, thin metal films may also be applied as the elastic structures by firstly depositing and patterning the layer and secondly suspending it from its support. Of particular interest are the interconnection layers in microelectronic circuits for applications in MEMS actuators. However, thin layers, such as nitride coatings, usually bear significant residual stresses that may reach into the GPa range calling for either stress-controlled design or the fabrication of actuators from bulk material. The situation is complicated by the fact that even the precise determination of elastomechanical constants of stressed layers is a challenging task and only few techniques are available.

2. Preparation of TiN Membranes

Figure 1a schematically shows the microchip architecture in a 0.25 μm CMOS/BiCMOS technology as fabricated in the IHP pilot line. Metal layers M1 to M4 essentially are made of Al:Cu exhibiting thicknesses from 0.5 to 2 μm and sheet resistances of some 10 mΩ sq⁻¹. The Al:Cu alloy is confined between TiN layers in order to suppress the oxidation of Al by reacting with SiO₂ from the interlayer dielectric (ILD) and to avoid the out-diffusion of Cu. The TiN layers of some 10 nm thickness are deposited by magnetron sputtering and show sheet resistances on the order of 10–30 Ω sq⁻¹; their deposition is usually preceded by a few nm Ti for adhesion improvement.

From the different metal layers used in the back-end-of-line (BEoL) stack, TiN appears of particular interest for BioMEMS devices, since it combines a high electrical conductivity, comparable to some metals, with a high resistance against biocorrosion. However, recent studies aiming at the usage of TiN in electrostatic driven MEMS were affected by the strong stress gradients within the layers and thus limited to actuator membranes with lateral dimensions in the sub-μm range (NEMS devices) requiring pull-in voltages in excess of 10 V.
Figure 1. Preparation of TiN membranes from BEoL architecture: a) schematics of IHP 0.25 μm CMOS/BiCMOS chip architecture with front-end-of-line (FEoL) and back-end-of-line (BEoL) stacks, the latter encompassing metal layers M1–M4 from TiN/Al:Cu/TiN, vertical tungsten interconnects, interlayer dielectrics from SiO2 (ILD) and a top passivation from SiO2/SiON (Pas); b) part of the process flow showing the preparation of a single TiN layer from a full metal TiN/Al:Cu/TiN stack; c) scanning electron micrograph (SEM) image of the BEoL cross section prepared by a focused ion beam (FIB) showing a part of the M3 stack reduced to the bottom TiN layer at a height of 2.5 μm above M1.

Various values for the elastic modulus $E$ of TiN can be found in the literature ranging from 427 to 590 GPa, where the precise value sensitively depends on preparation conditions.

A standard CMOS process flow was modified so as to allow for the integration of TiN membranes, but otherwise leave the standard BEoL stack unaffected. This goal was achieved by the process schematically depicted in Figure 1b. It can be seen from the scheme that the intended TiN membranes derive from the bottom TiN layer of a full metal TiN/Al:Cu/TiN stack of the standard flow. Only one additional resist mask is required in this approach that is used for the etching of top-TiN and Al:Cu in the areas intended for active MEMS structures. In these areas only a bottom-TiN layer remains, and any metal layer M1–M4 may be subjected to this treatment. Subsequently, the standard BEoL flow can be continued as usual by depositing the next ILD layer and planarizing by chemical–mechanical polishing (CMP). In this study, suspended TiN membranes were prepared from M3 layers in order to construct electrostatic actuators, in which the M1 layer would serve as counter electrode. Figure 1c shows a SEM cross section in the transition region of M3/TiN after BEoL processes were finished.

The formation of suspended TiN beams was performed by etching of embedding ILD layers with an HF-containing etchant and subsequent critical point drying (CPD) by supercritical CO2. This part of the full flow is schematically shown in Figure 2.

Voltage-dependent capacities may be designed by this process, in which the membrane and the M1 ground plate act as opposing electrodes and the TiN membrane may elastically deform, thereby varying the capacitance. A set of structures was fabricated as simply clamped beams with a width of $B = 6$ μm and differing lengths $L$ between 15 and 60 μm, see Figure 2b. These purely TiN-made beams were only $D = 50$ nm thick and the height above the M1 ground plate amounted to $h = 2.5$ μm. It may be recognised that the beams aligned parallel with the ground plate indicating a successful compensation of residual stress gradients.

3. Elastomechanical Constants

These model structures allowed for a determination of the elastic modulus $E_{\text{TiN}}$ by applying an electrical voltage and measuring the beam height in-situ by Doppler laser vibrometry. Figure 2c shows the bending monitored at the edge of the 30.3 μm beam, i.e., the time-resolved maximum deflection $z_{\text{max}}(t)$, when applying 10 V pulses at a rate of 10 kHz. Damped oscillations of the beam were then induced around both average positions

![Figure 2](image_url)

Figure 2. Suspension of TiN membranes and determination of elastic modulus from single clamped beams: a) part of the process flow showing the suspension of membranes or beams from the surrounding interlayer dielectric. b) Optical micrograph of suspended beams having a width of 6 μm and lengths of 15, 30, and 60 μm and a thickness of 50 nm; white scale bar 30 μm. c) $z_{\text{max}}(t)$ transient of the 30 μm beam without slits shown in (a), when pulses of 10 V were applied between the beam and the ground plate at a frequency of 10 kHz; measurements were performed by a laser Doppler vibrometer with a HeNe laser beam focussed on the middle of the beam (red hexagon); d) measured data points and non-linear regression curve from which the oscillation frequency $f = 84.2$ kHz and the elastic modulus $E_{\text{TiN}}$ were determined.
at 0 and 10 V, where the damping is caused by the interaction of the beam with the surrounding air, since the tests were performed under ambient laboratory conditions. The observed oscillation was assigned to the first fundamental mode of an excited beam\footnote{Excitation technique for thin metallic membranes.} having a frequency of $f = (0.162 D/L^2) \sqrt{E/\rho}$. The numerical value was determined by a non-linear regression of the signal shown in Figure 2d to amount to $f = 84.2$ kHz. An elastic modulus $E$ of the 50 nm TiN membrane of 476 GPa was derived from this result. Its precision is mainly determined by the uncertainty in mass density $\rho$ and thickness $D$ of the membrane, which can be estimated to be on the order of 4%.

An important presupposition for metallic membranes to be suitable for electrostatic actuation relates to the state of stress within them. Therefore, the layer architecture and processing was thoroughly adjusted to introduce a tensile stress into the TiN membranes in order to establish stable and reproducible beam deflections. The precise residual stress value $\sigma_{RS}$ was quantified by virtue of a double-clamped beam, compare Figure 3a. In this case, voltage pulses of 60 V were applied and deflection transients $z_{\text{max}}(t)$ in the middle of the beam were again monitored by laser vibrometry. Figure 3b displays the measured transient showing damped oscillations, which this time follow the fundamental mode of the vibrating string\footnote{Suitable fundamental modes for TiN membranes.} of frequency $f = \sqrt{\sigma_{RS}/4EI}$. It is realized from Figure 3c that the fitting by the model function yields an excellent agreement with measured data. Insertion of geometry and material parameters gives a residual stress of $\sigma_{RS} = 908$ MPa operative in the TiN membranes. The magnitude of this value appears rather high, albeit not uncommon in thin films.\footnote{Residual stress in thin films.} The positive sign, however, is remarkable, since it indicates a tensile stress that has successfully been introduced into the membranes and that contrasts with the compressive stresses usually observed in nitride coatings\footnote{Comparison of residual stress in nitride coatings.}.

It should be emphasized that the MEMS structures could be handled under usual laboratory conditions, although one might have expected particular precautions for the 50 nm TiN beams to be taken. Rather, they operated in reasonable measurement times for more than 100 cycles without showing signs of diminishing or failure. Among other reasons the mechanical stability was apparently provided by the built-in tensile strain and the supporting silicon substrate.

4. Performance of TiN Membranes in Microelectromechanical Structures

Figure 4d shows three- and fourfold-clamped beams that were prepared for electrostatic actuation tests. Again, their thickness and width are 50 nm and 6 $\mu$m, while they suspend 2.5 $\mu$m above the ground plate. The beams exhibit the shapes of X and Y structures with and without elongated slits. They all dispose of a mechanical spring in the middle of the structure in order to enable elastic bending in combination with a restoring force to increase with increasing deflection. For three of them (Xu, Yu, Xus) the spring takes the shape of a double-U, while the fourth (Xcs) has the form of two circles. In all cases, the width of the small spring beams was set by design to $b = 0.4$ $\mu$m and the gap between to 1.4 $\mu$m. After etching off ILD, the elastic relaxation in the suspended beams was observed to cause an expansion of the gap by 0.3–0.35 $\mu$m, which translates with the elastic modulus value given above to lateral strains on the order of 0.2%.

The total area $A$ of the multiple clamped beams exposed to the ground plate ranged between 1464 (Yu) and 1943 $\mu$m$^2$ (Xu). The electrical capacities of the structures may be approximated to first order by a parallel plate geometry, in which both electrodes are assumed to have the area $A$ of the suspended beam. The capacity values derived from this approach are denoted as projected capacities $C_{\text{proj}} = \varepsilon_0 A/h$ and lie between 5.2 und 6.9 fF. A precise determination, however, must also consider the stray capacitance between beam and ground plate. Based on finite-element simulations, values of 10.1, 13.1, 11.0 and 13.7 fF are then obtained for Yu, Xu, Xus, and Xcs structures, respectively.

Figure 4e shows the different $z_{\text{max}}(t)$ deflection curves that were measured for the cantilever structures when applying an increasing DC voltage between them and the ground plate. For comparison, the double-clamped beams from Figure 3a are also shown. In electrostatic actuation schemes, the deflection...
of the bendable electrode may proceed to a fraction of the initial electrode spacing, where the pull-in effect occurs. At this point, the attracting electrostatic forces become so large that the flexible electrode is drawn to the ground plate, which is usually accompanied by a plastic deformation and deterioration of the actuator. In case of the double-clamped beam, pull-in occurs at deflections of about \( h/3 \) of the initial spacing, \( z_{\text{pi}} = h/3 \). \[7\] But may take slightly different values for other geometries. In any electrostatic MEMS sensor, the measurement range advantageously encompasses the full swing of beam deflections between 0 and \( z_{\text{max}} \), to assure maximum sensitivity. It can be seen from Figure 4e that the double-clamped beams yielded noticeable deflections of some 100 nm only for voltages on the order of some 10 V. Such structures appear hardly suitable for sensor applications, when high sensitivities are required, difficult to achieve due to the high elastic modulus of TiN and the high tensile stress in the beams.

High sensitivities can be enabled, however, by including elastic springs as shown for the MEMS actuators in Figure 4a–d. It can be deduced from Figure 4e that deflections up to \( h/3 \) and beyond may be achieved already by applying small voltages of a few Volts. Capacitance changes \( \Delta C \) on the order of a 1–2 fF are seen to occur for the fourfold-clamped beams without pull-in to occur. Such variations \( \Delta C/C_{\text{proj}} \) are easily detected by common microelectronic circuits like a phase-locked loop (PLL) or derivates \[22\] with the capacitance change to be translated in a frequency shift. Quantifying the sensitivity \( \eta \) of sensors with variable capacitance by the relative change of capacity \( dC/C_0 \) per unit of voltage applied \( dV \), one arrives at \( \eta = (dC/dV)/C_0 \). It may then be concluded from the inspection of Figure 4e that sensitivities on the order of 5–20% \( V^{-1} \) may be realized with the fourfold-clamped TiN beams. This is a reliable for the full microelectronic integration, since CMOS devices usually operate for voltages of up to 3.5 V (depending on technology).

It can be concluded that ultrathin actuator membranes have been established, which appear particular suited for high-sensitivity BioMEMS applications. The development of a continuously monitoring in vivo glucose sensor operating by the principle of affinity viscosimetry has recently commenced. \[23\] Various other applications in the field of biotechnology for sensing of pressure, viscosity, etc., in microfluidic environments are to foreseen. In a modification of the architectural layer scheme presented here, the full metal stack may also be used for high-frequency MEMS applications like in radio-frequency switches. \[24\] In general, the design rules of TiN membranes allow their scaling to smaller dimensions by keeping the aspect ratio \( h/L \) constant, where \( h \) may even lie in the nm range. This opens the perspective for ultrathin TiN membranes to become a technological platform for the investigation and detection of nanoscopic effects and applications in general.

5. Conclusions

Summarizing, it has been shown that suspended TiN membranes may elegantly be prepared from the BEoL stack of a standard CMOS flow. An excellent compensation of stress gradients was achieved in combination with a tensile residual stress, \( \sigma_{\text{res}} > 0 \), rendering TiN beams and bridges suitable for electrostatic MEMS actuators. Precise elastomechanical
constants $\sigma_{RS} = 908$ MPa and $E = 476$ GPa were determined by Doppler vibrometry. In spite of the large elastic modulus, actuator structures could be prepared to operate in a varying capacitance scheme with pull-in voltages in the relevant CMOS range and sensitivities on the order of $5\text{–}20\% \text{ V}^{-1}$. The developed material module represents a technological platform for monolithic CMOS integration, enabling new MEMS and BioMEMS devices and their cost-effective mass production.

6. Experimental Section

Technological Preparations: MEMS structures with TiN membranes were fabricated within IHP’s CMOS/BICMOS pilot line on 200 mm CZ-Si wafers (p-type, 0.5 12m). See Figure 1b. Wafers were fully processed with modified and unmodified M3 layer stacks as outlined in the text and covered by photo resist. Areas intended for actuator cavities were illuminated in a photolithographic process with subsequent removal of exposed resist. Top SiON passivation and ILD layers, wherein the TiN beams with their small springs were embedded were wet etched with an HF containing solution, see Figure 2a. The high selectivity of the SiO$_2$ etchant towards TiN caused the etch process to stop at the top-TiN of the M1 ground plate. Unexposed resist was removed by bating the wafer in acetone and afterwards in iso-propanol. At this state of the process a drying of the wafer under ambient conditions would inevitable lead to sticking ("static friction") of the membrane to the ground plate causing its plastic deformation and destruction. The iso-propanol bath containing the wafer was therefore introduced into an 8" wafer critical point dryer (Automegasamdi 916B, Series C, Tousimis Research), where iso-propanol was displaced by supercritical CO$_2$ ($>31.1 {\text{deg} C}, >7.39$ MPa), transformed to ambient conditions and finally displaced by air. This enabled the preparation of suspended TiN membranes free from sticking to the ground plate.

Simulations: Finite element simulations were performed by virtue of ComSol 3.5a software package. The emes module for electrostatics calculations was applied to calculate the capacitances C of beam-ground plate configurations by solving Poisson’s equation.

Laser Doppler Vibrometry: The deflection of cantilever structures was measured by a laser Doppler vibrometer (Polytec MicroSystem Analyzer MSA-500). The system operates as a laser heterodyne interferometer, in which the HeNe laser spot (0.3 cm 2µm) was focussed on the centre of the cantilever. The frequency of the reflected laser beam is shifted by the Doppler effect due to the movement of the cantilever $f_0 \rightarrow f_0 + f_D$ with $f_D$ being analyzed by a modified Mach-Zehnder interferometer (Polytec Fiber Vibrometer OFV 552). The instrumental time and height resolutions amount to 10 ns and 15 pm, respectively. A wafer prober (Süss Microtech PA200) was installed to the instrument enabling full mappings of 200 mm wafers.

Acknowledgements

This study was performed within a BMBF funded project for the development of an implantable glucose sensor for diabetic patients (contract number 16SV3934). We thank our co-workers from IHP pilot line for thorough sensor preparations as well as J. Domke, R. Cernert, M. Gohike, J. Möller, F. Popiela, M. Richter, P. Schley, D. Schmidt, F. Tasdemir, and M. Ventzke for supporting electrical characterizations. Many thanks is also to J. Katzer for FB/SEM measurements, M. Wiestruck for helping in laser vibrometer testing, C. Kremn, A. Albrecht, and M. Hoffmann, TU Ilmenau, for first finite element simulations, S. Leidich, J. Mehner, TU Chemnitz, and D. Roscher for helpful discussions on MEMS operation. Moreover, we acknowledge the continuous support of G. Peine from Aktionszentrum BioTOP Berlin-Brandenburg.

Received: September 29, 2010
Published online: March 25, 2011