

Investigation of Mechanical Strength of Membrane Structure Consisting of Al/SiO₂/Al

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Abstract—Three-layer membrane structures consisting of Al/SiO₂/Al were fabricated using silicon group technology. The design of a research stand for determining the mechanical properties of membranes has been modernized. The values of mechanical strength and biaxial elastic modulus of the structures were obtained experimentally and calculated theoretically. Results of the developed model in the COMSOL Multiphysics proves a good correlation between the experimental critical pressure and the theoretical mechanical strength of the membrane. The distribution of mechanical stresses across the membrane using modelling and analytical calculation is shown. It is proved that the region of structure discontinuity is located at the membrane/substrate interface. The results obtained analytically, by modelling in the COMSOL Multiphysics and experimentally, were critically analyzed.

Keywords—mechanical strength; membranes; thin films; aluminium; silicon oxide; MEMS

I. INTRODUCTION

Membranes made by using micro- and nanotechnologies often plays a key role in sensors (MEMS devices). The membrane is a film consisting of one or more layers of different materials, which is located on the silicon substrate with a hole of different shape in the centre of it. The membrane can be part of anode in X-ray sources [1,2,3], which are a converter of electron energy into photon energy, or it can be part of a sensitive element in gas flow meters [4,5,6]. As the membrane area increases, the size of the focal spot and the intensity of the x-ray radiation also increases, the airflow sensors increase the working area of measurements. On the other hand, the mechanical strength of the structure is reduced, ergo, the amount of overpressure which membrane can withstand is also reduced. In addition, the likelihood of destruction increases when exposed to electrons from anode [7] or detecting high airflow velocity.

The mechanical properties of materials in micro-volume and macro-volume differ [8]. One of the ways to increase mechanical strength is to switch from a rectangular form of the membrane to a circular [9]. This is due to the fact that the destruction of the membrane in most cases occurs along the boundary of the membrane-substrate. The circle shape provides

significantly lower values of elastic deformations in comparison with rectangular shape of membrane, and strain values are evenly distributed along membrane contour [9]. Other factors of increasing mechanical strength include reduction of surface defects [10], change of the grain size of the structure materials [11], doping the film material with atoms of copper, zinc, magnesium, manganese, silicon [8].

II. FABRICATION OF STRUCTURE

A single crystal silicon wafer DSP12 (Boron doped) with a diameter of 150 mm with a crystallographic orientation of (100) and a thickness of 670 μm was used. A round membrane was formed on a square-shaped Si crystal with a side of 6 mm. The studied membrane structure consists of an upper aluminium layer with a thickness of 0.8 μm , a dielectric layer of SiO₂ with a thickness of 0.6 μm , and a lower aluminium layer with a thickness of 1.1 μm (Fig. 1).

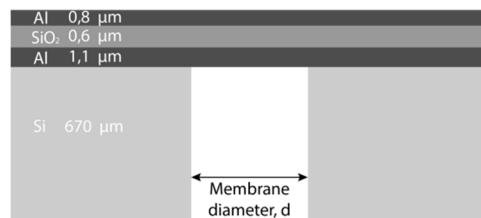


Fig.1. Schematic representation of the structure.

An aluminium film was formed by magnetron method from aluminium-silicon target. The SiO₂ layer was formed by PECVD method. The topology of the set of membranes represents a circle with a diameter of 1.0 mm and 1.4 mm, located in centre of crystal. There are no stress concentrators in the membrane due to use of round pattern for etching process.

This work was carried out on the equipment of the R&D center "MEMS and electronic components" and supported by Ministry of Education and Science of Russian Federation (within the agreement № № 075-15-2019-1650 (№ 05.594.21.0018)), id RFMEFI59419X0018).

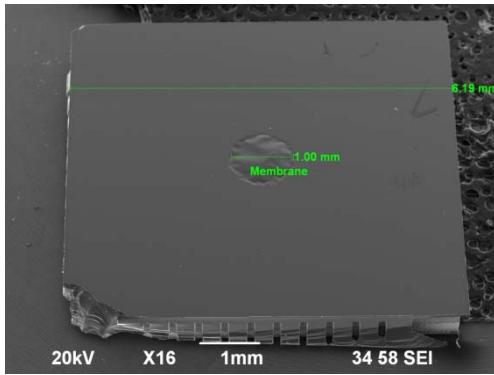


Fig.2. Picture of structure made with a scanning electron microscope.

III. ANALYSIS OF MECHANICAL STRENGTH OF STRUCTURE

In process of literary analysis, the following values of mechanical strength of thin aluminium composites and monolayers were found: from 0.04 to 0.08 GPa depending on grain size [11]; 0.2 GPa at an aluminum layer thickness of 373 nm and 0.55 GPa at a thickness of 205 nm [12]; 0.6 GPa at a thickness of 150 nm [13]; 9.0 GPa for the Al-12,6% Si composite film [10]. The following articles indicate the range of mechanical strength of a silicon oxide film: 0.364 ± 0.57 GPa PECVD SiO₂ 1.0 μm thick [14]; 0.89 ± 0.07 GPa of thermal SiO₂ [16]; from 1.2 to 1.9 GPa PECVD silicon oxide [15]; 8.4 GPa for filamentary structures of SiO₂ [17]. For further calculations, the mechanical strength of aluminium was taken 9.0 GPa and silicon oxide 8.4 GPa as a basis.

The theoretical value of mechanical strength (maximum mechanical stress) of the membrane σ_T is calculated by formula (1):

$$\sigma_T = \frac{\sigma_{\text{PolySi}} \cdot h_{\text{PolySi}} + \sigma_{\text{SiO}_2} \cdot h_{\text{SiO}_2} + \sigma_{\text{Si}_3\text{N}_4} \cdot h_{\text{Si}_3\text{N}_4}}{h_{\text{PolySi}} + h_{\text{SiO}_2} + h_{\text{Si}_3\text{N}_4}} \quad (1)$$

where $h_{\text{Al}1}$ is thickness of the lower layer of aluminium, h_{SiO_2} is thickness of silicon oxide, $h_{\text{Al}2}$ is thickness of upper layer of aluminium.

The calculated value of σ_T is 8.85 GPa. The distribution of mechanical stresses across the membrane is calculated by the formula (2) [8]:

$$\sigma = \frac{3 \cdot P}{8 \cdot r^2} * \sqrt{((1 + \mu)^2 \cdot (2a^4 - 8a^2r^2) + r^4 \cdot (10 + 12\mu + 10\mu^2))} \quad (2)$$

where a is radius of membrane, h is thickness of membrane, P is pressure on membrane, μ is Poisson's ratio of membrane, r is distance from centre of membrane.

The results of the calculation according to formula (2) of the distribution of mechanical stresses along the diameter of the membrane are shown in Fig. 3. According to an analytical calculation, the greatest mechanical stress σ_{\max} arises when the distance from the centre (middle) of the membrane is equal to the radius, i.e. $r=a$. Thus, the predicted critical overpressure value P_{pr} is calculated by formula(3)[8]:

$$P_{pr} = \frac{\sigma_{\max} \cdot h^2}{a^2 \cdot B(\mu)} \quad (3)$$

Coefficient $B(\mu)$ is calculated by formula (4):

$$B(\mu) = \frac{3}{4} \sqrt{1 + \mu^2} \quad (4)$$

The value of Poisson's ratio of the membrane μ is calculated similarly to the approach as in formula (1). Given that μ_{Al} is 0.34 and μ_{SiO_2} is 0.2, Poisson's ratio of the membrane μ will be 0.3. Therefore, value of the coefficient $B(\mu)$ is 0.78. Thus, according to the calculations by formula (2) for a membrane with a radius of 0.5 mm, the predicted value of the critical overpressure P_{pr} is 2.82 atm, and for a radius of 0.7 mm - 1.44 atm.

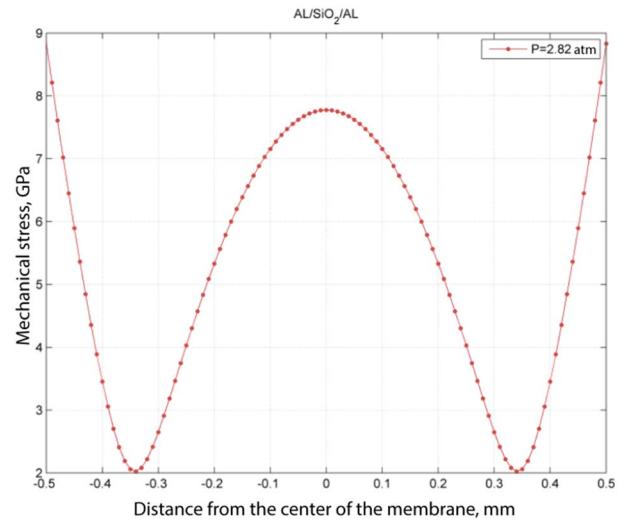


Fig.3. The distribution of mechanical stresses in a membrane with a radius of 0.5 mm using formula (2).

There also was compiled a model in COMSOL Multiphysics. The coordinate regions of the substrate are from -700 to -500 μm and from 500 to 700 μm along the X-axis. The membrane region is located symmetrically with respect to the coordinates of the $X = 0$ axis. The membrane layers were formed with a rectangular grid, which consists of 2240 elements along the X-axis (grid pitch 1.6 μm) and 30 elements along the Y-axis (each film layer is divided into 10 elements). In the region of silicon substrate, a free triangular mesh type was selected. The dependence of the distribution of mechanical stresses on a membrane with a diameter of 1.0 mm and in a thin film at an excess pressure of 5.8 atm (Fig.4) obtained from experimental data is obtained.

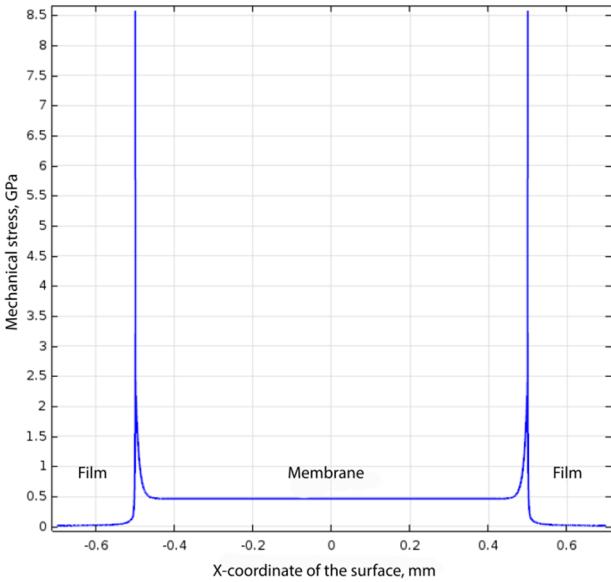


Fig.4. The distribution of mechanical stresses in a membrane with a radius of 0.5 mm using COMSOL Multiphysics model

The maximum mechanical stress is localized at the membrane/substrate interface. The developed model in the COMSOL Multiphysics correlates well (relative error of 3%) with experimental and theoretical data (at an excess pressure of 5.8 atm, the maximum value of mechanical stresses is 8.57 GPa). There is no membrane in the silicon cavity (Fig. 4) of the structure after the critical deformation of the membrane. Thus, the membrane breaks at the membrane/substrate interface. In (Fig. 5), we also can see an absence of membrane material in the silicon cavity after critical deformation of membrane. Thus, membrane breaks at the membrane/substrate interface.

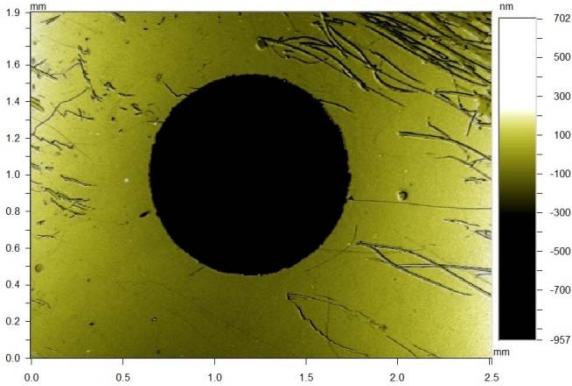


Fig.5. Top view of structure after it's breaking.

IV. ANALYSIS OF THE BIAXIAL MODULUS OF ELASTICITY OF THE STRUCTURE

By analyzing the dependence (formula (5)) of the deflection of the membrane w from the excess pressure P , we can determine the biaxial elastic modulus $E/(1-\mu)$:

$$P = C_1 \cdot \frac{\sigma_0 \cdot h \cdot w}{a^2} + C_2 \cdot \frac{E \cdot h \cdot w^3}{(1-\mu) \cdot a^4} \quad (5)$$

where P is the excess pressure, σ_0 is residual mechanical stresses in the structure at $P = 0$, h is the thickness of the membrane, w is deflection of the membrane, and a is radius of membrane, E is Young's modulus, and μ is Poisson's ratio.

The values of the coefficients C_1 and C_2 depend on the shape of the membrane. Typically, when working with round membranes, $C_1 = 4$ and $C_2 = 8/3$ are used [10]. The dependence $P(w)$ can be divided into a steep and gentle region. The steep region criterion is satisfied for small values of the deflection of the membrane w , i.e. the first term is much larger than the second. The value of the biaxial elastic modulus $E/(1-\mu)$ is calculated on the flat region of dependence (5) for large values of the deflection of the membrane W , i.e. the value of the first term can be neglected (formula (6)):

$$\frac{E}{1-\mu} = \frac{P \cdot a^4}{C_2 \cdot h \cdot w^3} \quad (6)$$

Young's modulus of aluminium is 70 GPa [18], Young's modulus of silicon oxide is also 70 GPa [16]. Therefore, Young's modulus of the membrane is 70 GPa. The theoretical value of biaxial elastic modulus of the membrane $E/(1-\mu)$ will be 100 GPa with a Poisson's ratio of the membrane μ equal to 0.3, calculated earlier.

The dependence of the maximum deflection of the membrane from excess pressure is presented below (Fig. 6). In formula (5), the value of residual stresses in the structure is 100 MPa. To increase correlation between the calculation by formula (5), modelling in a COMSOL Multiphysics and experimental data, the initial deflection (at $P=0$) is 10 μm in the simulation and 20 μm in the analytical calculation, and the value of the biaxial elastic modulus $E/(1-\mu)$ is 50 GPa.

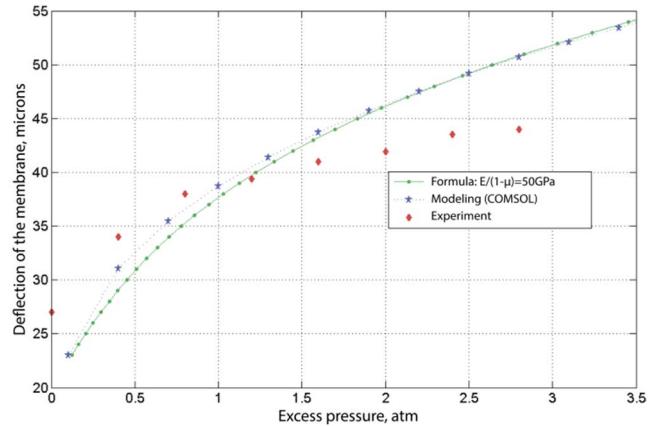


Fig.6. The dependence of the maximum deflection of the membrane from excess pressure for membranes with a radius of 0.5 mm.

Several conclusions can be drawn from Figure 5. The initial structure has a significant amount of deflection of the membrane, which is not taken into account in the original formula and in the COMSOL Multiphysics model. Calculating the elastic modulus from formula (5) from experimental data in P range from 1.2 to 2.8 atm, we obtain a set of values from 36.7 to 61 GPa, which is several times less than the theoretical value. This may be due to the fact that the experimentally measured array of values of w (P) is located in a steep

region of dependence (4). Therefore, it is necessary to develop accessories to protect the profilometer from the material of the exploding membrane during scanning. This will make it possible to obtain a larger data array $w(P)$, which will lead to an increase in the accuracy of the determination of the biaxial elastic modulus.

V. EXPERIMENTAL MEASUREMENT OF MECHANICAL STRENGTH

To determine the mechanical properties of the membrane elements, a previously developed stand was modernized [8]. Excessive pressure is supplied from the magistral (instead of the compressor). Thus, the upper pressure range is expanded to 6.5 atm, and the stability of the pressure value in the system is also increased.

The critical overpressure (excess pressure) values experimentally determined on the upgraded stand. At a diameter of 1.0 mm, the mechanical strength of the Al/SiO₂/Al membrane is 5.8 ± 0.4 atm for a diameter of 1.0 mm (15 samples) and 3.8 ± 0.5 atm for a diameter of 1.4 mm (18 samples). It is noticeable that the results obtained are highly reproducible. The experimental value of the mechanical strength of a three-layer membrane is 18.2 GPa.

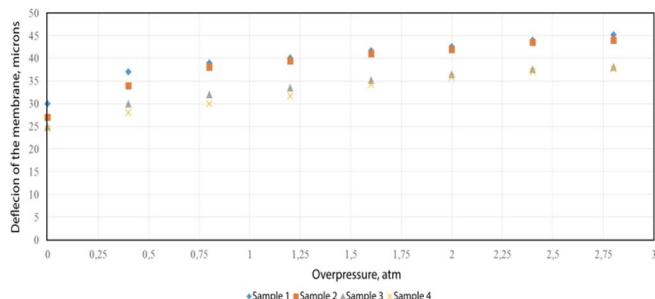


Fig.7. Experimental dependence of membrane deflection on overpressure.

During experiment at excess pressures of more than 3 atm, deflection of membrane was not measured (Fig. 6). This is due to the protection of an expensive profilometer lens from tearing membrane material. The rupture of the membrane can occur during the scanning of the surface by the profilometer.

VI. CONCLUSIONS

The experimental value of the mechanical strength of a three-layer Al/SiO₂/Al membrane is 18.2 GPa, the biaxial elastic modulus $E/(1-u)$ is 50 GPa. The critical excess pressure of Al/SiO₂/Al membrane structures on a silicon substrate is 5.8 ± 0.4 atm for a radius of 0.5 mm and 3.8 ± 0.5 atm for a radius of 0.7 mm. The result allows you to use these membranes as anodes of x-ray sources of the cross-type type with a margin of mechanical strength several times. Discrepancy between the analytical calculation data, modelling and experimental results is explained by the effect of using a set of alternating layers instead of a monolayer of the membrane; the effect of increasing the mechanical strength of the films due to the modernization of the deposition technology; the effect of increasing adhesion between the silicon substrate and the silicon atoms present in the lower layer of aluminum obtained from the aluminum-silicon target, magnetron method.

REFERENCES

- [1]. . Kilpatrick et al. X-ray limits on the progenitor system of the Type Ia supernova, Monthly Notices of the Royal Astronomical Society, 481 (3), pp. 4123-4132, 2018, DOI: 10.1093/mnras/sty2503
- [2]. D. Stupple, V. Kemp, M. Oldfield, J. Watts and M.Baker. Modeling of Heat Transfer in an Aluminum X-Ray Anode Employing a Chemical Vapor Deposited Diamond Heat Spreader. Journal of Heat Transfer, 140 (12), 124501, 2018, DOI: 10.1115/1.4040953
- [3]. B. Kozioziemski et al. An x-ray optic calibration facility for high energy density diagnostics. Review of Scientific Instruments, 89 (10), 10G112, 2018, DOI: 10.1063/1.5038742
- [4]. Y. Kessler, B. Ilic, S. Krylov and A. Liberzon. Flow Sensor Based on the Snap-Through Detection of a Curved Micromechanical Beam. Journal of Microelectromechanical Systems, pp.945 – 947, v. 27 , Issue 6, 2018, DOI: 10.1109/JMEMS.2018.2868776
- [5]. M. Horning, R. Shakya and N. Ida. Design of a Low-Cost Thermal Dispersion Mass Air Flow (MAF) Sensor. Sensing and Imaging, 19 (1), 21, 2018, DOI: 10.1007/s11220-018-0204-0
- [6]. M. Islam et al. A prominent smart gas meter, 2nd International Conference on Electronics, Materials Engineering and Nano-Technology, IEMENTech, v.27, issue 6, 2018, DOI: 10.1109/JMEMS.2018.2868776
- [7]. E. Sugata, H. Yokoya and K. Kitakaze. The mechanical strength of thin films under electron bombardment. Journal of Electronmicroscopy, vol.7, p.19-20, 1959, DOI: 10.1007/978-3-662-01991-7_116
- [8]. Gusev, E. E., Borisova, A. V., Dedkova, A. A., Salnikov, A. A., Kireev, V. Y. The Effect of Ion Beam Etching on Mechanical Strength Multilayer Aluminum Membranes. 2019 IEEE Conference of Russian Young Researchers in Electrical and Electronic Engineering (EIConRus), DOI:10.1109/eiconrus.2019.8657243
- [9]. A. Vlasov, T. Civinskaya and A. Shahnov. Analysis of the influence of the shape of the membrane on the mechanical strength and stability of the parameters of MEMS pressure sensors. MES-2016 (in Russian)
- [10]. M.G. Mueller, M. Fornabaio, G. Zagar, A. Mortensen, Microscopic strength of silicon particles in an aluminium-silicon alloy. Acta Materialia, Volume 105, 15 February 2016, pp. 165-175, DOI: 10.1016/j.actamat.2015.12.006
- [11]. Ramnath Venkatraman and John C. Bravman, Separation of film thickness and grain boundary strengthening effects in Al thin films on Si. J. Mater. Res., Vol. 7, No. 8, pp. 2040-2048, Aug 1992, DOI: 10.1557/JMR.1992.2040
- [12]. A. Boe et al.. MEMS-based microstructures for nanomechanical characterization of thin • lms. Smart Materials and Structures, 18 (2009) 115018 (8pp), DOI: 10.1088/0964-1726/18/11/115018
- [13]. N. Andre. Microfabrication-based nanomechanical laboratory for testing the ductility of submicron aluminium • lms. Microelectronic Engineering, Volume 84, Issue 11, November 2007, pp. 2714-2718, DOI: 10.1016/j.mee.2007.05.039
- [14]. W.N. Sharpe et. al.. Strain Measurements of Silicon Dioxide Microspecimens by Digital Imaging Processing. Experimental Mechanics, 2007, 47(5), pp.649-658, DOI 10.1007/s11340-006-9010-z
- [15]. Toshiyuki Tsuchiya, Atsuko Inoue, Jiro Sakata. Tensile testing of insulating thin films; humidity effect on tensile strength of SiO₂ films. Sensors and Actuators, v.82, pp.286-290, 2000, DOI: 10.1016/S0924-4247(99)00363-5
- [16]. Jinling Yang, Fracture Properties of LPCVD Silicon Nitride and Thermally Grown Silicon Oxide Thin Films From the Load-De•ection of Long Si₃N₄ and SiO₂/Si₃N₄ Diaphragms. Journal of Microelectromechanical Systems, vol. 17, no. 5, october 2008
- [17]. K. Petersen. Silicon as Mechanical Materials. Proceedings of the IEEE, 1982, v. 70, issue: 5 , pp.420 – 457.
- [18]. Mei-Ni Sua, Ben Young. Material properties of normal and high strength aluminium alloys at elevated temperatures. Thin-walled structures, v.137, pp.463-471, 2019, DOI: 10.1016/j.tws.2019.01.012