Design of silicon membranes for ultrasonic transducers and its fabrication by anisotropic wet etching

S V Malohatko1, E Yu Gusev1, J Y Jityaeva2 and O A Ageev1,2

1Department of Nanotechnologies and Microsystems, Southern Federal University, Taganrog 347922, Russia
2Research and Education Center “Nanotechnologies”, Southern Federal University, Taganrog 347922, Russia

e-mail: malohatko@sfedu.ru

Abstract. The aim of this work is to calculation and fabrication silicon membranes for acoustic sensors with operating ranges of resonant frequencies from 10 kHz to 100 MHz and pressures from 0.1 to 10³ kPa. In this paper, analytical dependences of pressure and resonance frequency on geometrical parameters of membranes are presented. The ranges of the thickness (30-50 µm) and the length of the edge membranes (0.2-1.0 mm) were defined. Experimental studies of the etching of the silicon wafer with a solution of 30% KOH were carried out and the etching rate was found to be 1.2-1.8 µm/min. Anisotropic wet etching was used to form square-shaped silicon membranes with a thickness of 30-50 µm and an edge length of 0.2 to 1.0 mm with resonant frequencies in the range of 0.7 to 30 MHz. The obtained results can be used in the development of acoustic sensors based on monocrystalline silicon.

1. Introduction
Acoustic sensors operating in the frequency range from tens of kilohertz to hundreds of megahertz have found wide application in various fields of industry and medicine, for example, such as ultrasonic location and actuating devices [1,2], visualization of internal organs [3–6] and other. At the same time, not single sensors, but their arrays integrated with electronic systems become popular [5,7]. In the last twenty years, there has been an expansion of the range of tasks solved by such systems, and as a result, requirements for increasing the measurement range, accuracy, reducing size and power consumption are increasing. The noted tendency determined the need for group manufacturing of such sensors using techniques and technologies of micromechanical processing [6-8]. The micromechanical design of acoustic transducers has allowed achieving a number of advantages, such as versatility, low noise and power consumption, as well as the ability to integrate with elements of semiconductor microelectronics [9]. Solving the latter problem, along with the choice of materials and technological processing conditions of operations, the size of the acoustic part of the device, in particular, the sensitive element, becomes important. A sensitive element of a micromechanical acoustic sensor is a membrane whose geometric dimensions and materials are determined at the design step taking into account the field of application. For acoustic sensors used in ultrasonic devices, pressure values range from 0.1 to 10³ kPa. To operate the device in the megahertz range, the size should be about micrometers [10]. Researchers use a variety of materials and optimize the structures of the membrane for a particular frequency or a narrow range [1-12]. Thus, it becomes necessary to manufacture an array of membranes with different geometric parameters in a single technological route.
that will reduce the cost and obtain membranes with uniform quality and range of operating frequencies. Such membranes are traditionally made of monocrystalline silicon or by bulk micromachining [6–12]. One of the most common group of bulk micromachining is anisotropic silicon etching, which includes deep reactive ion etching, plasma and wet etching [13]. The advantages of the latter are accessibility and cheapness. Traditionally, the structure is etched with solutions of TMAH, EDP and KOH [13]. In the case of KOH, etching is carried out at 10-50% and a temperature of 50-90°C [14]. Studies have shown that etching in 10-20% KOH solution leads to an increase in surface roughness [14]. When the concentration increases to 40%, the etched surface becomes smoother, but etching rate of silicon dioxide protective film increases, which often leads to defects in the structure [14]. So, the optimization of the geometry of membrane in the array to obtain the desired frequency and pressure ranges for ultrasonic devices, and optimization of the conditions for their fabrication, are important and relevant.

The purpose of the work is to calculate the design and to form silicon membranes for acoustic sensors with operating ranges of resonant frequencies from 10 kHz to 100 MHz and pressure from 0.1 to 10^3 kPa by anisotropic wet etching in KOH solution.

2. Theoretical and experimental details

The design of the membrane in the form of a thinned silicon wafer of a square shape was calculated on the basis of the well-known expressions (1), (2) for the mechanical characteristics of pressure \( p \) and resonance frequency \( f_0 \) of hard wafers with initial internal stresses [15]:

\[
p = C_1 \frac{E h^3}{(a/2)^4(1-v^2)} b + C_2 \frac{\sigma_0 h}{(a/2)^2} b + C_3 \frac{E h}{(a/2)^4(1-v^2)} b^3, \tag{1}
\]

here \( E \) – Young modulus, \( v \) – Poisson ratio, \( C_1, C_2, C_3 \) – coefficients depending on the shape of the membrane, \( \sigma_0 \) – internal stress, \( h, a, b \) – thickness, edge length and the deflection of the membrane;

\[
f_0 = \frac{1}{2\pi} \left( \frac{k}{m} \right)^{1/2}, \tag{2}
\]

here \( k, m \) – the stiffness and mass of the membrane.

The membrane thickness and edge length of the varied from 10 to 100 µm and from 0.1 to 1.0 mm, respectively. The geometrical parameters of the membrane are selected taking into account technique features of mechanical characteristics [16] measurement based on optical determining the deflection of the membrane for the particular case of the 10 nm.

Monocrystalline silicon wafer (100) with a thickness of 320 µm were used as substrates. Thermal oxide (SiO\(_2\)) films with a thickness of 600±20 nm were used as a protective layer on the front and back of the substrate during wet etching. After cleaning, several samples were covered both sides with 1 µm thick SiO\(_2\) films as additional protection layers by plasma enhanced chemical vapor deposition [17,18]. Then, the oxide layer was patterned on the back side of the substrate using contact photolithography and isotropic wet etching [18,19]. At the final step, anisotropic wet etching of silicon was performed in 30% KOH solution at 70°C to a depth of 270-290 µm, followed by releasing from the protective layers of silicon oxide in 10% HF solution. The etching depth was controlled by stylus profilometry.

3. Results and discussion

The effect of the geometric parameters of the membrane on the pressure that ensures its deflection at 10 nm is shown in figure 1. Figure 1 shows that the influence of the considered geometric parameters on the load pressure has a polar direction: both the thickening of the membrane and a decrease in the length of its edge leads to a pressure shift towards higher values. The observed trend is also true for the frequency dependence (see figure 2). Thus, for applications that are focused on low pressures at a relatively high level of resonant frequency, a compromise solution will need to be found.
The dependence of the geometric parameters of the membrane on the resonant frequency, according to expression (2), is shown in figure 2.

The analysis of the estimated dependences shows that the optimal geometric parameters that meet the acceptable values of pressure and resonant frequency are thickness 50 µm, the length of the membrane edge from 0.2 to 1.0 mm.

Anisotropic wet etching was chosen to form membranes, a process flow was proposed, topology calculations were performed, and template containing determined values of geometrical parameters of windows was designed and obtained.

Arrays of SiO$_2$/Si(100)/SiO$_2$ and SiO$_x$/SiO$_2$/Si(100)/SiO$_2$/SiO$_x$ structures with open "windows" in the protecting layer were formed and etched (figure 3). According to experimental data, the rate of silicon etching was 1.2-1.8 µm/min, which is close to [19,20]. The total etching time to a depth of 270 µm was 215 min. At the same time, in the first structures, it was observed that the rate of oxide etching was 4 nm/min and the oxide was completely removed after 150 min, so the desired depth was not reached. In structures with plasma oxide, the average and local residual thickness of the oxide was 1000 nm and 930-950 nm, respectively. In this way, the thickness of the plasma oxide could be safely reduced to 500-600 nm.

As a result, a series of square silicon membranes was formed with a thickness of 50 µm and edge lengths ranging from 0.2 to 1.0 mm (figure 4), which is potentially capable of providing sensor operation in the frequency range from 10 kHz to 100 MHz.
4. Conclusion
The paper considers approaches to evaluating the design of monocrystalline silicon membranes obtained by anisotropic wet etching. The dependences of pressure and resonant frequency on the geometric parameters of the membrane at a fixed deflection level are calculated. The ranges of thickness (30-50 µm) and membrane edge length (0.2-1.0 mm) corresponding to the values of pressure (0.1-10³ kPa) and resonant frequency (10 kHz-100 MHz) typical for acoustic sensors used in ultrasonic devices are determined. The template topology is calculated. An experimental study of anisotropic wet etching of monocrystalline silicon in a solution of 30% KOH at 70°C was performed. The etching rate of silicon was 1.2-1.8 µm/min and the etching rates of thermal and plasma oxides were 4 nm/min and 3.2 nm/min, respectively. It is experimentally established that the optimal thickness of the protective oxide layer in such etching conditions should be at least 1.1 µm, including 500 nm of plasma oxide.

Figure 3. The schematic diagram showing the cross-sectional view of non-released membrane. Figure 4. Optical image of an array of silicon membranes

An array of square-shaped silicon membranes with a thickness of 50 µm and edge lengths from 0.2 to 1.0 mm, designed for a load of 0.1-10³ kPa with resonant frequencies in the range from 0.7 to 30 MHz, was formed to create acoustic sensors applied for the ultrasonic range. The formed structures will be further studied using a fiber-optic adaptive holographic interferometer [16].

The obtained results could be used in the development of acoustic sensors, microelectronic sensors and micromechanics based on monocrystalline silicon membranes.

Acknowledgment
The research is supported by the Russian Science Foundation (project № 18-29-11019 and project № 19-37-90139). The results were obtained using the equipment of Research and Education Center “Nanotechnologies” of Southern Federal University.

References
[1] Drinkwater B W, Wilcox P D 2006 NDT E Int. 39 525
[2] Jiang X, Kim K, Zhang S, Johnson J, Salazar G 2013 Sensors 14 144
[3] Watson B, Friend J, Yeo L 2009 Sens. Actuators A Phys. 152 219
[4] Fenster A, Downey D B 1996 IEEE Eng. Med. Biol. Mag. 15 41
[5] ter Haar G R 2001 Echocardiography 18 317
[6] Qiu Y et al. 2014 Sensors 14 14806
[7] Wu J, Fedder G K, Carley L R 2004 IEEE J. Solid-State Circuits 39 722
[8] Hautefeuille M, O’Mahony C, O’Flynn B, Khalfi K, Peters F 2008 Microelectron. Reliab 48 906
[9] Judy J W 2001 Smart Mater. Struct. 10 1115
[10] Muralt P, Baborowski J 2004 J. Electroceram. 12 101
[11] Wang Z, Zhu W, Tan O K, Chao C, Zhu H, Miao J 2005 Appl. Phys. Lett. 86 033508
[12] Muralt P, Ledermann N, Paborowski J, Barzegar A, Gentil S, Belgacem B, Petitgrand S, Bosseboeuf A, Setter N 2005 *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* **52** 2276

[13] Mamilla V R, Chakradhar K S 2014 *Procedia Materials Science* **6** pp 1170–1177

[14] Yun M 2000 *Journal of the Korean Physical Society* **37** 605

[15] Alvi P A 2012 *MEMS Pressure Sensors: Fabrication and Process Optimization* (Barcelona: IFSA)

[16] Romashko R V, Bezruk M N, Kamshilin A A, & Kulchin Y N 2012 *Quantum Electronics* **42**(6) 551

[17] Gusev E Y, Jityaeva J Y, Avdeev S P, Ageev O A 2018 *Journal of Physics: Conference Series* **1124** 0220345

[18] Gusev E Yu, Jityaeva J Y, Ageev O A 2018 *Materials Physics and Mechanics* **37** 67

[19] Ageev O A, Konoplev B G 2019 *Nanotechnology in microelectronics* (Moscow: Nauka)

[20] Kirt R W, Muller R S 1996 *Journal of microelectromechanical systems* **5** 256