ARCHIVES OF ACOUSTICS
25, 4, 479–486 (2000)

FABRICATION OF THE MEMBRANES ON A SILICON BASE FOR THE SENSORS WITH THE ULTRASONIC WAVES LAMBA-TYPE GENERATED BY USING THE INTERDIGITAL TRANSDUCERS

J. GOŁĘBIOWSKI

Technical University of Łódź
Institute of Theoretical Electrotechnics,
Metrology and Materials Science
(90-924 Łódź, Stefanowskiego 18/22, Poland)
e-mail: jacekgol@ck-sg.p.lodz.pl

The construction of the microsensor with the ultrasonic wave of Lamba-type as well as the conditions of the wave propagation are presented. The possibilities of the fabrication of multi-layer membranes on a silicon base using the microelectronics technologies are presented and discussed. The analysis of the usefulness of the processes mentioned for the production of thin membrane sensors was carried out taking into consideration intrinsic stresses. A summary of the experimental results is given and the most useful parameters of the membrane ultrasonic sensors are pointed out.

1. Sensor construction and conditions of Lamba wave propagation

The sensor membrane with the Lamba type ultrasonic wave consists of several layers of different thickness. For a membrane made of a silicon plate, the basic layer, which determines the elasticity properties of the whole membrane, is a monocrystalline silicon layer or a layer of silicon nitride or oxide. The others are piezoelectric and metallic layers. In the metallic layer, the interdigital electrodes are made of the piezoelectric transducers (IDT). A view of the transducers that are placed on the membrane surface as well as the contact fields (white field denotes the surface etched in the silicon — at the bottom) is presented in Fig. 1. The principle of the sensor work consists in the generation of an ultrasonic wave in the plane membrane by means of two interdigital transducers (IDT). One of them works as a transmitter and the other one as the receiver. The Lamba waves are determined as transverse waves which propagate in the plate plane. Their polarization plane is normal to the plane of the plate. These waves are symmetric and asymmetric in relation to the plane in the middle thickness of the membrane. Each of those waves can contain different numbers of the mode vibrations denoted by the indices 0, 1, 2. The velocity of the Lamba wave propagation depends on the wave frequency and the membrane thickness [2, 3].

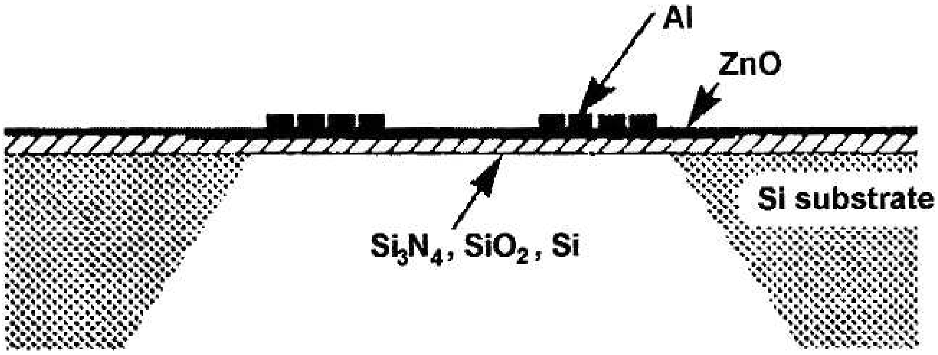

Fig. 1. The interdigital piezoelectric transducers (Al) on the surface of the sensor membrane.

For the symmetric wave it is possible to obtain lower phase velocities and a lower wave frequency in the sufficiently thin membrane. In order to obtain high sensitivities, in the application of membrane sensors as mass detectors, thin membranes should be used [1]. That is why for the following conditions should be fulfilled by the membrane: the maximum wave amplitude should be smaller than the thickness d, the wave length λ should be significantly larger than the membrane thickness ($\lambda \gg d$), the membrane width a and length b should be significantly larger than the thickness ($a \gg d$, $b \gg d$). The technologies of the production of thin elastic layers in the sensors' membranes are described below. The following layers are deposited on the base: a piezoelectric layer which is made of zinc oxide (ZnO) and a metallic one made of aluminium (Al) [4].

2. Fabrication of thin silicon membranes

The silicon membranes were obtained by anisotropic etching of the silicon. Wafers with the diameter of 3″, made of the monocrystalline silicon one-sidedly polished with the orientation of < 100 > and a donor conductivity, phosphorus doped with resistivity of 1 − 2 Ωcm, were the basic material. The thickness of the wafers was 380 μm. The upper surface of the wafers was protected by silicon nitride against etching in potassium hydroxide (KOH). This layer was made by the LPCVD (Low Pressure Chemical Vapour Deposition) method in the reaction of dichlorosilane (SiH_2Cl_2) with ammonia (NH_3) at a temperature of 800° C; the ratio of SiH_2Cl_2 to NH_3 was 4 : 1. The thickness of the silicon nitride layer (Si_3N_4) was 20 nm [6]. The membrane shape on the bottom side of the silicon wafer was defined by a photolitographic process. The silicon nitride layer from the membrane area was etched off in a plasma process and then the photoresist was removed. Finally the wafer was etched in a aqueous solution of KOH. As the result of the anisotropic silicon etching, membranes with sizes of 2×2.5 mm and the thickness of 12 − 15 μm were obtained [5]. The cross-section of the sensor membrane with the silicon layer is shown in Fig. 2.

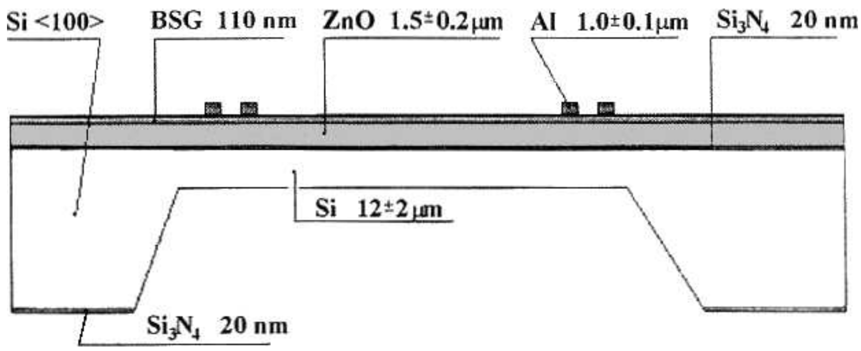

Fig. 2. The cross-section through the sensor membrane with the monocrystalline silicon layer: ZnO — the zinc oxide layer, Al — the aluminium layer, Si_3N_4 — the silicon nitride layer, Si — the wafer made of <100> monocrystalline silicon.

3. Fabrication of the silicon nitride membranes

The silicon nitride Si_3N_4 layer was made by the LPCVD method at a temperature of 800°C by the reaction of dichlorosilane (SiH_2Cl_2) with ammonia (NH_3) [11]. The ratio of the dichlorosilane to the ammonia was 4 : 1. The purity of the gases used in the process was "electronic grade". Under the described conditions, the deposition rate of the Si_3N_4 layer was about 0.066 nm/s. The one-side polished silicon wafers with the diameter of 3″ and thickness of 380 µm were the substrate for the silicon nitride. They were made of monocrystalline silicon of n-type conductivity (phosphorus doped). The resistivity of the wafers and their crystallographic orientation were $1-2\,\Omega$cm and $< 100 >$, respectively. The silicon nitride layer of a thickness of about 1.3 µm was deposited during 330 min. The thickness was measured by a reflectometer with accuracy of ±20 nm. The refractive index of this layer was about 2.1. For the stoichiometry of the silicon nitride layer, the refractive index was equal to 2.0. The influence of the deposition conditions on the value of the refractive index in the silicon nitride layer is shown in Fig. 3.

As it can be seen, all the nitride layers obtained are not of the stoichiometric composition. The latter is Si_xN_y layer with $x > 3$ and $y < 4$. The parameters of the deposition process were chosen so as to minimize the stresses in the deposited layer. The total stresses, which are the sum of the thermal stresses and the intrinsic one, were lower than $8 \cdot 10^8$ Pa [7]. The photomasking process was done at the bottom side of the wafers and the Si_3N_4 was etched in a plasma process. Then, the wafers were etched in a KOH solution. From of the anisotropic silicon etching, membranes made of silicon nitride with sizes of 2×2.5 mm and a thickness of 1.3 µm were obtained. In the membranes, however, too high intrinsic stresses were present. They caused cracking of the membranes. The dependence of the total stresses and the intrinsic one in the silicon nitride layer on the DCS/NH_3 ratio is shown in Fig. 4.

It was obvious that even for low values of the total stresses, which are the sum of the thermal stresses and intrinsic one, those components could reach high values. It is

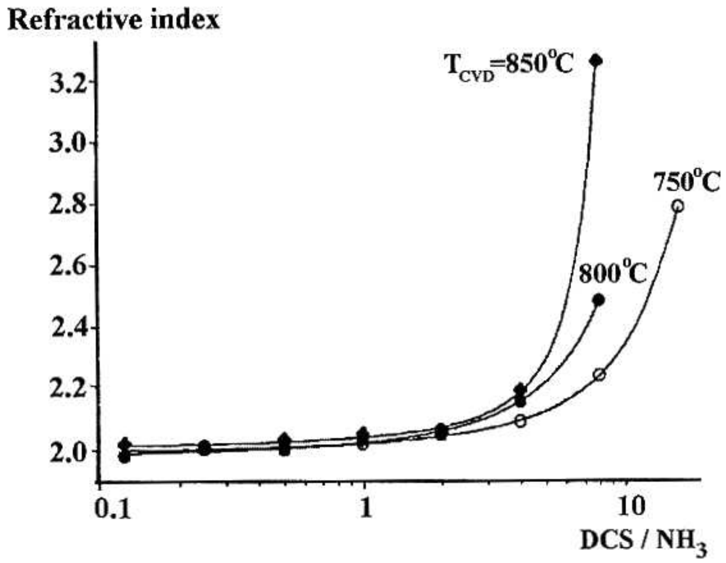

Fig. 3. The dependence of the refractive index on the DCS/NH_3 ratio.

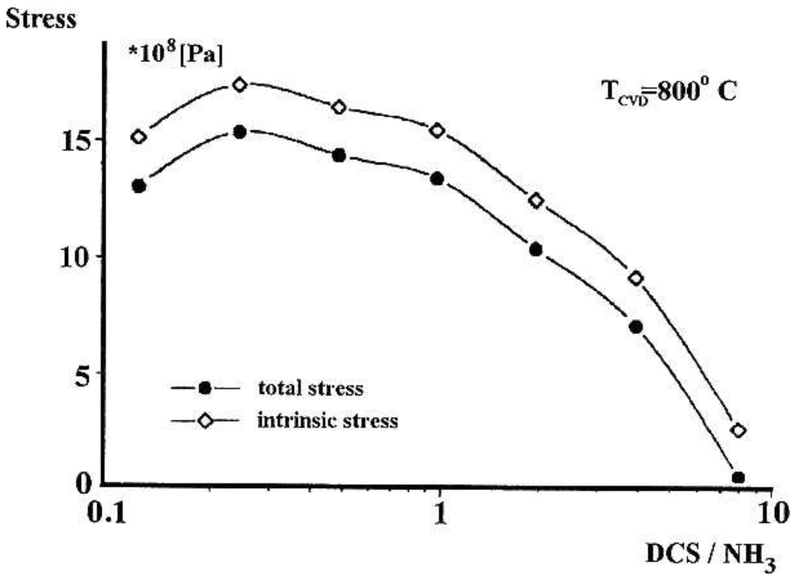

Fig. 4. The dependence of the total and intrinsic stresses on the DCS/NH_3.

the result of the existence of the stresses of different signs. Then, the silicon layer was removed in the etching process. The thermal stresses were removed during etching the silicon layer under a silicon nitride membrane. The remained intrinsic stresses were too high, thus they destroyed the thin silicon nitride layer.

A photograph of the membrane surface with silicon nitride is shown in Fig. 5. Traces of the cracks can be seen on the surface.

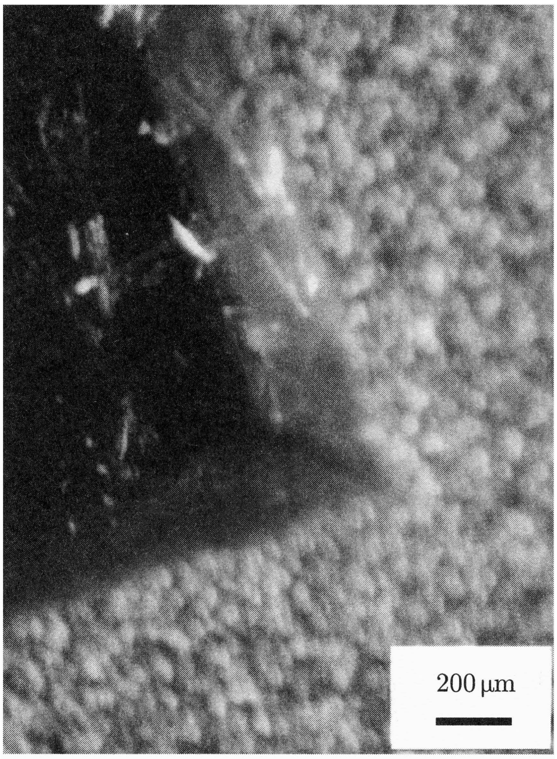

Fig. 5. Micrograph of the membrane surface with a silicon nitride layer (view from the etched surface side).

4. Fabrication of the Boron-Silicon-Glass membrane

The boron doped silicon dioxide layer (BSG-Boron Silicon Glass layer) was deposited by the APCVD (Atmospheric Pressure Chemical Vapour Deposition) method by the reaction of silane (SiH_4), boron hydride (B_2H_6) and oxygen at 300°C. The process of deposition was carried out in the VAPOX 5000 reactor at three different concentration of boron hydride in silane: 1, 2 and 3 percent. The volume ratio of oxygen to the mixture of silane and boron hydride was 2 : 1 [10]. There was atmospheric pressure in the deposition area. Under those conditions, the deposition rate of BSG was about 20 nm/s. The purity of the gases were of "electronic grade". The thickness of the BSG layers obtained was 2.15 μm. The BSG layer was deposited on silicon wafers coated with a thin silicon nitride layer. The thickness of Si_3N_4 was 20 nm; it was deposited by the LPCVD method. Then the second silicon nitride layer 20 nm thick was deposited on the top of the BSG layer.

Silicon nitride layers deposited from both the sides of the BSG layer protected the latter during the silicon etching. The silicon nitride layers were very thin and, therefore, their influence on the BSG membrane deformation was insignificant. The thickness of the layers was measured by a reflectometer and ellipsometer. The photomasking process was performed on the bottom of the wafers and next the silicon nitride layer was etched by plasma. Finally, the silicon was etched in a KOH solution. Membranes of sizes of 2×2.5 mm and the thickness of about 2.1 µm, consisting of a BSG and two Si_3N_4 layers, were obtained by the anisotropic etching. The dependence of total stresses in the BSG layer on the concentration of the boron doping is shown in Fig. 6. The membrane with the BSG layer is presented in Fig. 7.

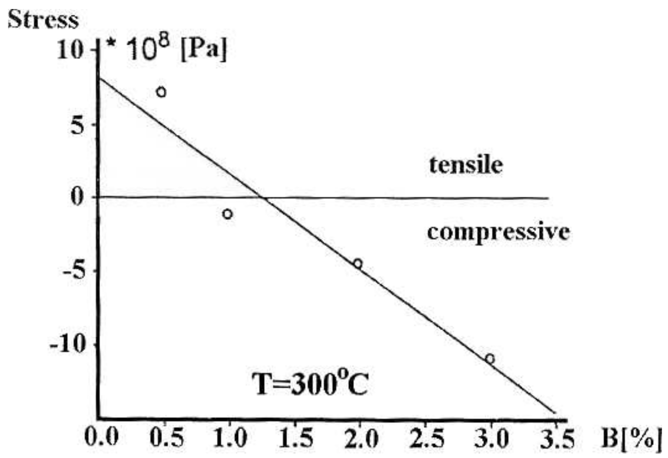

Fig. 6. The dependence of the total stresses on the concentration of the doped boron.

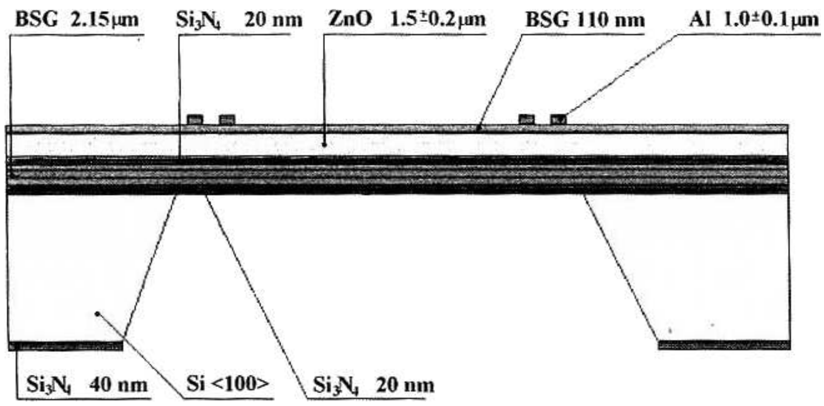

Fig. 7. The cross-section through the sensor membrane with the BSG layer: ZnO — zinc oxide, Al — aluminium, Si_3N_4 — silicon nitride, BSG — boron silicon glass, Si — monocrystalline <100> silicon.

A photograph of the membrane surface with a BSG layer with apparent surface deformations is shown in Fig. 8.

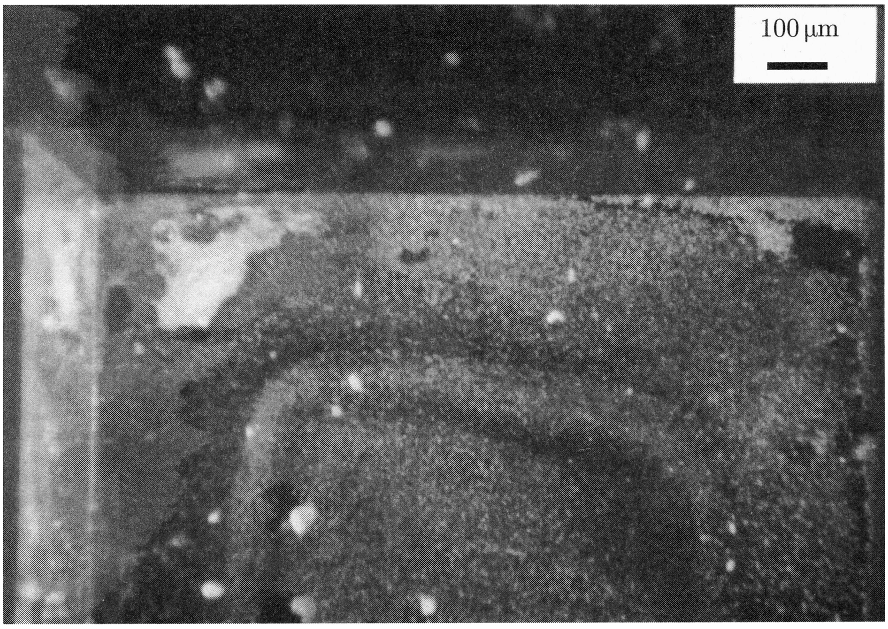

Fig. 8. The micrograph of the membrane surface with a BSG layer (view from the etched surface side).

5. Conclusions

The investigations of the fabrication of thin membranes made of monocrystalline silicon, silicon nitride and boron silicon glass lead to the following conclusions:

1. The rectangular silicon membranes with the sizes (length, width) of several millimetres (the membranes produced were 2.5×2 mm in size) and a thickness ranging from ten to nineteen micrometers meet the requirements of sensors with a Lamba wave. The ratio of the membrane thickness to its other sizes, however, is too low ($1:150 - 1:250$) to obtain high sensor sensitivities.

2. The rectangular silicon nitride membranes with the sizes (length, width) of several millimetres and the thickness ranging from one to several micrometers were damaged (cracked) because of the high intrinsic stresses. However, it is possible to obtain membranes of sizes (length, width) lower than one millimetre and a thickness below one micrometre. These small membranes can be applied in the sensors with Lamba wave. Thus allows the ranging of the ratio thickness to other sizes from $1:500 - 1:1500$ and the obtaining of highly sensitive sensors. In this case the paths of electrodes in the IDT transducer should not exceed $3 - 5$ μm.

3. It is possible to obtain rectangular boron silicon glass (BSG) membranes with sizes (length, width) of a few millimetres and thickness ranging from one to two micrometres. The lowest total stresses appear at a concentration of boron hydride in silane of about 1% (Fig. 5). However, surface deformations on some of the membranes were observed; those deformations disqualify them as membranes in sensors with Lamba wave. The production of BSG layers has not been worked out yet and needs further investigations.

Apart from the processes that were discussed in this paper, it is possible to manufacture thin membranes in a different ways, for example, by using etch-stop technique with diffusion or an epitaxial layer. These layers should have a high boron concentration in silicon (more than $8 \cdot 10^{19}\,\mathrm{cm}^{-3}$) [12].

Acknowledgements

This work was sponsored by the Polish State Committee for Scientific Research, grant No. 8T10C02216. The author also thanks Dr P. Grabiec from Institute of Electron Technology in Warsaw for her help in carrying out the investigations.

References

[1] J. Gołębiowski, *Silicon membrane sensors based on piezoelectric resonators*, Proceedings of the 6 MIXED Design of Integrated Curcuits and Systems MIXDES'99, Krakˇw, 285–288 (1999).

[2] R. White, P.J. Wicher, S.W. Wenzel and E.T. Zellers, *Plate-mode ultrasonic oscillator sensors*, IEEE Trans. Ultrasonics Ferroelectr. Freq. Control, **34**, 162–171 (1987).

[3] D.F.L. Jenkis, M.J. Cunningham, G. Velu and D. Remiens, *The use of sputtered ZnO piezoelectric thin as brod-band microactuators*, Sensors and Actuators, A**63**, 135–139 (1997).

[4] J. Gołębiowski, *Fabrication of piezoelectric thin film of zinc oxide in composite membrane of ultrasonic microsensors*, J. Mat. Sci., **34**, 19, 4661–4664 (1999).

[5] R.T. Howe and R.S. Muller, *Stress in polycrystalline and amorphous silicon thin films*, J. Appl. Phys., **54**, 4674–4678 (1983).

[6] M. Chang, J. Wong and D.N.K. Wang, *Low stress, low hydrogen, nitride deposition*, Solid State Technology, **5**, 462–468 (1988).

[7] E.A. Irene, *Residual stress in silicon nitride films*, J. Electr. Mater., **5**, 287–297 (1976).

[8] M. Sekimoto, H. Yoshihara and T. Ohkubo, *Silicon nitride single-layer x-ray mask*, J. Vac. Sci. Technol., **21**, 1017–1021 (1982).

[9] A. Muller *et al.*, *Dielectric membrane support*, European Semiconductor, **19**, 27–29 (1997).

[10] J. Gołębiowski, T. Budzyński, P. Grabiec, J. Jaźwiński and J. Koszur, Proceedings of the Conference on "Optoelectronic and Electronic Sensors COE'98", Jastarnia, Poland, 239–243 (1998).

[11] B. Folkmer, P. Steiner and W. Lang, *Silicon nitride membrane sensors with monocrystalline transducers*, Sensors and Actuators, A**51**, 71–75 (1995).

[12] Ch. Burrer and J. Esteve, *High-precision BESOI-based resonant accelerometer*, Sensors and Actuators, A**50**, 7–12 (1995).