# Investigation of micro mechanic membranes with functional piezoelectric film

# V. Lendraitis\*, G. Seniūnas\*\*, V. Snitka\*\*\*, V. Mizarienė\*\*\*\*, R. Barauskas\*\*\*\*\*

\*Kaunas University of Technology, Studentų 65, 51369 Kaunas, Lithuania, E-mail: vitas.lendraitis@ktu.lt

\*\*Kaunas University of Technology, Studentų 65, 51369 Kaunas, Lithuania, E-mail: gediminas.seniunas@ktu.lt

\*\*\*Kaunas University of Technology, Studentų 65, 51369 Kaunas, Lithuania, E-mail: vsnitka@ktu.lt

\*\*\*\*Kaunas University of Technology, Studentų 65, 51369 Kaunas, Lithuania, E-mail: vimiz@ktu.lt

\*\*\*\*\*Kaunas University of Technology, Studentų 50, 51368 Kaunas, Lithuania, E-mail: rimantas.barauskas@ktu.lt

## 1. Introduction

PbTiO - PbZrO (PZT) solid solutions comprise the class of the materials, which are characterized by the exceptional ferroelectric and piezoelectric properties. Nowadays the intensive investigations of thin coating application of those materials on the surface and volumetric acoustic wave devices, sensors and actuators in micro system technology, submicron measurement memory devices etc., are performed all over the world. The coatings for these devices must be marked by low roughness and grain size, as well as high coefficients of piezoelectric module and electro mechanic connection. The sol-gel technology, used for such thin piezoelectric coatings, has many advantages: the possibility to manage exactly tachometry and microstructure, low cost, relatively low temperature of the process, the possibility to get the coatings on big surfaces and good repetition of the coating parameters. The technology could be matched with the processes of micro system technologies [1, 2].

The aim of the investigation is to test the sol-gel piezoelectric film (PF) technology on Si membranes and to decide on the possibility to apply such micromechanical membranes in micro object nano positional devices.

# 2. Modeling of micro mechanic membrane, applying finite elements method

For realization of the aim, the nanoposition actuator prototype – sandwich structure, made of vibroactive piezoelectric film (PF), put on the Si substrate, applying the sol-gel technology, and Pt electrodes system, enabling to realize the PF excitation in various membrane places – was developed and tested. (Fig. 1, Table 1).



Fig. 1 Construction of micromechanical membrane

The application of Si for PF substrate was determined by wide possibilities of three-dimensional structure formation (etching) in Si substrates and applying photolithography technologies, essential for further modifications of mechanical membrane construction, as well as technical requirements for nanoscanner actuators, - high frequency of free oscillations (kHz line), minimal transmission function hysteresis, transmission function stability regarding time and work cycles, slight temperature errors. The Si substrates fully meeting these requirements are used in micro electronics.

Substance	E, GPa	$v \qquad \frac{\rho}{10^3 \text{ kg/m}^3}$		<i>h</i> , μm
Pt	146.9	0.39	21.45	0.1
PZT	86.2	0.287	7.75	0.9
Si (membrane)	120	0.42	2.33	40
Si (substrate)	120	0.42	2.33	500

Substance characteristics of micromechanical membrane

Table 1

Note: *E* is Young module; *v* is Poison coefficient;  $\rho$  is substance density; *h* is substance thickness.

While performing the modeling of tested construction applying finite elements method (FEM), the structure is devided into finite elements of simple geometry, the type of which depends on the solved task. Mechanical, electrical and piezoelectric correlations are given in the form of matrix equations, in which the structure depends on the would-be equation system. Every element is described by its form functions  $N_i$ , which determine the node interrelation and are common to displacements, mass and potentials. The form functions describe mechanical and piezoelectric phenomena, evaluating their interaction by one of the independent variable systems.

Applying the selected law and after deviding the tested structure into finite elements of particular size, the node coordinate is defined, which is necessary for the form functions  $N_i$ , from which the constructions for matrix displacements

$$\begin{bmatrix} N^{\phi} \end{bmatrix}^{T} = \begin{bmatrix} N_{1} & 0 & 0 & \dots & N_{n} & 0 & 0 \\ 0 & N_{1} & 0 & \dots & 0 & N_{n} & 0 \\ 0 & 0 & N_{1} & \dots & 0 & 0 & N_{n} \end{bmatrix}$$
(1)

and potentials

$$\begin{bmatrix} N^{\phi} \end{bmatrix}^{r} = \begin{bmatrix} N_{1} & N_{2} & \dots & N_{n} \end{bmatrix}.$$
 (2)

The mass matrix of the structure, the volume of which is V and density  $\rho$  is expressed as

$$\left[M\right] = \int_{V} \rho \left[N^{U}\right] \left[N^{-U}\right]^{T} dV$$
(3)

In the piezoelectric body, where mechanical and electric potential forces act, the equation system for one element or the whole structure is expressed as (4)

$$\begin{bmatrix} [M] [0] \\ [0] [0] \end{bmatrix} \begin{cases} \{u\} \\ \{\Psi\} \end{cases}^{+} \begin{bmatrix} [C] [0] \\ [0] [0] \end{bmatrix} \begin{cases} \{u\} \\ \{\Psi\} \end{bmatrix}^{+} \\ \begin{bmatrix} [K] \\ [K^{-z}]^{T} \\ [K^{-d}] \end{bmatrix} \begin{bmatrix} \{u\} \\ \{\Psi\} \end{bmatrix}^{-} = \begin{cases} \{F\} \\ \{Q\} \end{bmatrix}$$
(4)

where u,  $\Phi$  are respectively, matrixes of node shifts and node potentials; F, Q are respectively, vectors of mechanical and potential forces; C is friction, viscosity or loss matrix; K is resilience matrix.

In general case

$$\begin{bmatrix} K \end{bmatrix} = \int_{V} \begin{bmatrix} B_{u} \end{bmatrix}^{T} \begin{bmatrix} C \end{bmatrix} \begin{bmatrix} B_{u} dV \end{bmatrix}$$
(5)  
$$\begin{bmatrix} B_{u} \end{bmatrix} = \begin{bmatrix} \frac{\delta}{\delta x} & 0 & 0 \\ 0 & \frac{\delta}{\delta y} & 0 \\ 0 & 0 & \frac{\delta}{\delta z} \\ \frac{\delta}{\delta z} & 0 & \frac{\delta}{\delta z} \\ \frac{\delta}{\delta y} & \frac{\delta}{\delta x} & 0 \\ 0 & \frac{\delta}{\delta z} & \frac{\delta}{\delta y} \end{bmatrix} \begin{bmatrix} N^{u} \end{bmatrix}^{T}$$
(6)

where  $\begin{bmatrix} B_u \end{bmatrix}$  is operator.

Losses are evaluted by the friction matrix

$$[C] = \alpha[M] + (\beta + \beta_c)[K] + \sum_{k=1}^{Nel} [C_k]$$
(7)

where  $\alpha$  is mass matrix factor;  $\beta$ ,  $\beta_c$  are respectively, constant and dependent on frequency matrix factors;  $[C_k]$  is (4) viscosity matrixes of dependence shape elements.

Having the answer of equation system (4), the interpretation of the results is relatively simple. Vibration distribution on the surface of the piezoelectric structure is derivable directly from the node displacement matrix. The piezofilm admittance Y, considering that the piezoelement excitation is made by 1 V voltage, and after harmonic analysis, is found, integrating the calculated induced loads in the whole electrode area, i.e.

$$Y = \omega \int_{A} Q_{mi} = \omega \sum_{n} Q_{mi}$$
(8)

where  $\omega$  is frequency;  $Q_{mi}$  is node loads; A is electrode area; n is number of nodes in the electrode area.

For the analysis of the micro mechanical membrane, two types of finite elements were selected – Solid 5 and Solid 45, which are usually used for the modeling of electric field in three-dimensional space and for the constructions of resilient substances. The electric field is created between the bottom and top Pt electrodes (Fig. 1).

In case of symmetrical membrane for faster calculations the quarter of the sample was modeled (Fig. 2).



Fig. 2 Quadrant model of the micromechanical membrane at the excitation of 10V voltage in the centre of the membrane: a - first mode; b - second mode

The membrane was excited applying 5V and 10V voltage, changing the position of electrodes at the short and long sides. The modeling was performed through the excitation in the centre and the perimeter of the membrane. The calculations made at the frequency range of 0.01 - 175 kHz showed, that the highest amplitude, as predicted, was obtained exciting the centre of the membrane (Fig. 3).

#### 3. Methodology investigation

During the investigation piezoelectric modules  $d_{33}$ , based on four types of micro mechanic membrane films PbTiO<sub>3</sub> with different alloys were evaluated: homogeneous structures, such as PbSm<sub>0.05</sub>TiO<sub>3</sub> (PST), Pb<sub>1.0175</sub>Sm<sub>0.05</sub>(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub> (PSZT), Pb(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub> (PZT at the morph tropic phase range), and the heterogeneous structure PST/PSZT.

The films were made applying the sol-gel technology and putting 8 layers (in case of heterogeneous structure: 4 PST and 4 PSZT layers) on Si substrate. The film is formed using centrifuge method and spreading the sol layer on the surface, then it is heated at 700°C. The film thickness reaches 700-900 nm.



Fig. 3 Amplitude-frequency dependence, determined by FEM:  $A_{fem}$  - oscillation amplitude; f - excitation frequency; U -excitation voltage; 1, 2, 3 - respectively, excitation in the centre of the membrane at the long and short sides

The structure bases were made of Pt/Ti/SiO<sub>2</sub>/Si 19x15x0.5 mm substrate, in the centre of which the 6.2x4.3x0.04 mm membranes were etched. The piezoelectric film was formed on the Si substrate plane, working surface of which was firstly polished and chemically cleaned, then covered by oxide (4 hours in the oxidation furnace at 1200°C). Thickness of the Pt electrode layers (Fig. 1) is 100 nm. Top Pt electrodes in the membrane centre and near the sides of the membrane were covered using vacuum steaming method, after the formation of PZT 52/48 film had been performed (10 layers, thickness about 900 nm). In case of putting many layers of piezoelectric compositions, tension between the layers may cause micro cracks, which can greatly increase electric conductivity between the bottom and top electrodes, at the same time decreasing the effective signal. To avoid this, on one of the samples additional 100 nm thickness TiO<sub>2</sub> layer was put between PF and top electrodes. After covering and performing preliminary PF testing, the films were polarized, using the corona poling method. Polarization duration was 30 min, the voltage - 15kV, temperature - 250°C.

#### 4. Investigation results

Piezoelectric module was determined by evaluating the excited vibration parameters in the films with the laser Doppler vibrometry system, made of Polytec OFV353



Fig. 4 Experimental setup scheme for laser Doppler vibrometry measurements

sensor head, OFV3001 control unit, DSP synchronic amplifier AMETEK 7225 and generator HP3325<sup>a</sup> (Fig. 4). The film vibration amplitude correlations with of the size signal magnitude are presented in Fig. 5. The determined values of the PF film piezoelectric module are given in Table 2.



Fig. 5 Displacement  $A_e$  of the film surface as a function of the amplitude of driving voltage U: 1 - PZT poled; 2 - PZT unpoled; 3 - PSZT poled; 4 - PSZT unpoled; f - frequency of excitation

·	Table 2
PF film piezoelectric module $d_{33}$ evaluation resu	lts

Piezoelectric module	Film				
	PST	PSZT	PZT	PST/PSZT	
$d_{33}$ , pm/V, unpoled samples	16	7	93	10	
$d_{33}$ , pm/V, poled samples	7	62	419	35	

Very high coefficient of the polarized PZT film is noted. Though this value is much higher, than noted in many sources, it almost coincides with 400 pm/V [3]. This unexpectedly high piezoelectric activity PZT at the range of morph tropic rhombic-etragonal phase is explained by the fact that elongation follows in the direction of monoclinous crystal deformation, which forms during the polarization process [4].

When the polarization vector of ferroelectric domain has normal component to the surface and electric voltage is switched on, depending on the direction of the poled vector and the reverse piezoelectric effect, the domain and piezoelectric geometry change their orientation. Exciting the Si membrane with PF alternating voltage, its vibration amplitude is proportional to piezoelectric module of the film and the phase is proportional to the domain orientation.

Dynamics PF mechanic deformation of the Si membrane was tested in the measurement system of atomic force microscope (AFM), switching the voltage between the conductive cantilever probe in contact with PF and the bottom Pt electrode (Fig. 6, 7).

For testing, the AFM microscope, designed at KTU Scientific Centre of Microsystems and Nanotechnologies, with NT-MDT controller and branded software with additional original programs, developed at the scientific centre, was used.

The micro mechanical membrane testing are performed to determine the application possibilities of Si



Fig. 6 Scheme of experimental setup for piezoelectric force microscopy investigations



Fig. 7 General view of vibration measurement stand: *1* - AFM measurement head; *2* - observation microscope with video camera; *3* - AFM controller BI022MT; *4* - screw for the sample positioning in X-Y plane; *5* - lock -in-amplifier; *6* - DC power supply HY3002-2; *7* - low frequency generator G3-112

membrane with PF functional film for nanopositioning of micro objects. Fig. 8 shows the amplitude – frequency correlation with PZT film obtained during the registration of surface vibration amplitudes in the centre and the edge of membrane. The exciting 10 V signal was put between the bottom electrode and 1 mm<sup>2</sup> electrodes, vaporized in



Fig. 8 Experimental amplitude - frequency characteristics of micro mechanical membrane with PZT functional film:  $A/A_{max}$  - comparative vibration amplitude; f excitation frequency; U - voltage of excitation; I, 2 - respectively, excitation in the centre and the long side of the membrane

The experimental investigation confirms the FEM results of micro mechanical membrane modeling and enables to define the further optimization direction of the membrane - actuator construction, evaluating the importance of biologic object investigation, applying scanning microscopy and at the same time the necessity of expanding the scanning range by fixing the membrane not to the whole perimeter, but through the internal friction flexures, formed on the Si substrate. Such membrane alternators with functional piezo electric coating are produced applying laser technologies and are distinguished by small size, high resonance frequency, minimal stroke/voltage characteristic hysterics and low costs. They could be used for the positioning of micro objects (e.g. AFM cantilever, biological sample) at the nanometric accuracy in the micrometric shift range. They are compatible with the commercial AFM operation software, while the range of positioning and resonance frequency are determined during the designing process, selecting the inner friction geometry of joints.

#### 5. Conclusions

1. The work of micro mechanical membrane in the vibroactuator mode was modeled applying the Finite Elements method.

2. The technology for forming  $PbTiO_3$ -PbZrO<sub>3</sub> functional films on Si membrane was developed.

3. During the investigation piezoelectric values  $d_{33}$  of different film were quantifiable and the most perspective for nanopositioning piezoelectric film - PZT was found.

4. Applying AFM system, the dynamic characteristics of micro mechanical structures with piezoelectric films were evaluated.

5. The positive investigation results of micro mechanic membranes with functional PF film allow optimizing the membrane construction and designing the scanning microscopy nanopositioning devices.

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V. Lendraitis, G. Seniūnas, V. Snitka, V. Mizarienė, R. Barauskas

# MIKROMECHANINIŲ MEMBRANŲ SU FUNKCINIU PJEZOELEKTRINIU SLUOKSNIU TYRIMAS

#### Reziumė

Straipsnyje nagrinėjama galimybė panaudoti Si membranas su funkciniu pjezoelektriniu sluoksniu mikroobjektams nanopozicionuoti.

Baigtinių elementų metodu nustatytos mikromechaninės membranos pagrindinės rezonansinės modos bei jų priklausomybė nuo žadinimo vietos membranos paviršiuje. Pateikta PbTiO<sub>3</sub>-PbZrO<sub>3</sub> dangų nusodinimo ant Si substrato ir poliarizavimo technologija, įvertinti dangų pjezomoduliai. Naudojant atominių jėgų mikroskopo sistemą, nustatytos sukurtų mikromechaninių struktūrų dinaminės charakteristikos.

Tyrimo rezultatai naudotini kuriant naujo tipo skenuojančios mikroskopijos nanopozicionavimo sistemas.

V. Lendraitis, G. Seniūnas, V. Snitka, V. Mizarienė, R. Barauskas

# THE INVESTIGATION OF MICRO MECHANIC MEMBRANES WITH FUNCTIONAL PIEZOELECTRIC FILM

# Summary

In the study the possibility of using Si membranes with functional piezoelectric film for the nanopositioning of micro objects is discussed.

Applying the Finite Elements method, the main

resonance modes of micro mechanical membrane and the dependence on the location of excitation on the surface of the membrane are determined. The PbTiO<sub>3</sub>-PbZrO<sub>3</sub> film coating on the Si substrate and the polarization technology are presented, the film piezomodules are evaluated. Applying the atomic force microscope system, the dynamic characteristics of michromechanic structures created, are determined.

The results of the investigation could be applied in developing a new type of nanopositional systems of scanning microscopy.

В. Лендрайтис, Г. Сянюнас, В. Снитка, В. Мизарене, Р. Бараускас

# ИССЛЕДОВАНИЕ МИКРОМЕХАНИЧЕСКИХ МЕМБРАН С ФУНКЦИОНАЛЬНЫМ ПЬЕЗОЭЛЕКТРИЧЕСКИМ ПОКРЫТИЕМ

#### Резюме

В статье рассматривается возможность использования мембран из кремния (Si) с функциональным пьезоэлектрическим покрытием для позиционирования микрообъектов с нанометрической точностью.

Методом конечных элементов определены основные резонансные моды мембраны и их зависимость от места возбуждения на поверхности мембраны. Представлены основы технологии покрытия субстратов из кремния пьезоэлектрическими растворами PbTiO<sub>3</sub>-PbZrO<sub>3</sub> с последующей их поляризацией, определены пьезомодули покрытий. При помощи системы микроскопа атомных сил определены динамические характеристики созданных микромеханических структур.

Результаты исследования могут быть использованы при создании нанопозиционирующих устройств нового типа для сканирующей микроскопии.

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