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Investigation of micromechanical gas sensor response

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1. Introduction

With the invention of the atomic force microscope (AFM), micromechanics and the microscopy of nanometer objects became closely related. The AFM systems sensors – micro cantilevers, giving the possibility to measure interatomic forces within the range of piconewtons, can be used not only for the evaluation of surface characteristics, but also as autonomous micro sensors for the transformation of various signals into the microcantilever mechanical movement with the resolution power reaching up to angstrom parts.

The use of the microcantilever structures for gas detection is determined by the need to increase the sensors' response and accuracy, and the possibility, using simple and cheap construction, to perform quantitative gas components evaluation of the environment tested [1].

This study analyses the dependence of two-layer microcantilever dynamic characteristics on chemical composition of detected environment, and the possibility to make complex material analysis device on a single chip, using micromechanical sensor units.

2. Microcantilever mathematical model

The microcantilever is designed using a stand-fixed, tough, constant geometry, rectangle cross-section rod (Fig. 1).

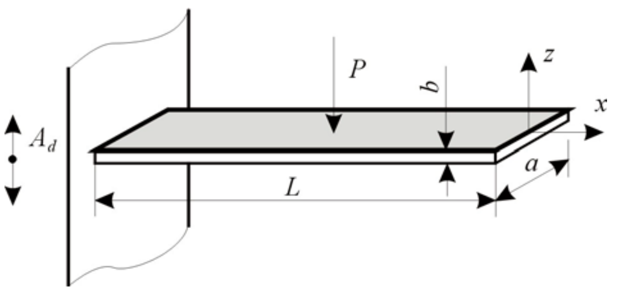

Fig. 1 Scheme of the microcantilever mathematical model

The oscillation of the rod, which is tightly fixed at the one end of the base and loose at the other one, (considering, that the coefficient of environment viscosity damping is constant) will be described as partial function of the fourth row differential equation [2]

$$EI\frac{\partial^4(z,t)}{\partial x^4}+\rho S\frac{\partial^2 z(x,t)}{\partial t^2}-\gamma\frac{\partial z}{\partial t}=P \qquad (1)$$

where E and ρ are respectively, the Young module and density of the rod material; I is moment of inertia (for the rectangle cross-sectional rod $I=a\cdot b^3/12$, a and b are respectively, width and thickness of the rod); $S=a\cdot b$ is cross-sectional area; γ is viscosity damping coefficient of the environment, in which the rod oscillates; P is load, affecting the entire rod area.

In case, when the rod oscillations are excited by the base harmonic oscillations $A_d sin\ \omega t$, the load corresponds to the rod inertia force, and the Eq. 1 can be changed to [2]

$$EI\frac{\partial^4(z,t)}{\partial x^4}+\rho S\frac{\partial^2 z(x,t)}{\partial t^2}-\gamma\frac{\partial z}{\partial t}=\rho S\omega^2 A_d\, sin\,\omega\, t \qquad (2)$$

The common type solution of this equation is obtained using the method of variable separation and presented as

$$z(x,t)=Z(x)T(t)=$$
$$=\left[AS_1(\beta x)+BS_2(\beta x)+CS_3(\beta x)+\right.$$
$$\left.+DS_4(\beta x)\right]sin\left(\omega t+\varphi_0\right) \qquad (3)$$

where

$$\beta=\sqrt[4]{\frac{\rho S\omega^2-i\gamma\omega}{EI}} \qquad (4)$$

$$S_1(\beta x)=\frac{1}{2}\left(cosh\,\beta x+cos\,\beta x\right)$$

$$S_2(\beta x)=\frac{1}{2}\left(sinh\,\beta x+sin\,\beta x\right)$$

$$S_3(\beta x)=\frac{1}{2}\left(cosh\,\beta x-cos\,\beta x\right)$$

$$S_4(\beta x)=\frac{1}{2}\left(sinh\,\beta x-sin\,\beta x\right)$$

here A, B, C and D are constants; φ_0 is initial phase; β is coefficient evaluating number of wave; $S_i(\beta x)$ is Krylov functions; $i=\sqrt{-1}$.

Making use of Krylov functions' properties, the part of solution $Z(x)$, called the form of oscillations, could be presented as

$$Z(x)=Z(0)S_1(\beta x)+\frac{Z'(0)}{\beta}S_2(\beta x)+\frac{Z''(0)}{\beta^2}S_3(\beta x)+$$
$$+\frac{Z'''(0)}{\beta^3}S_4(\beta x) \qquad (5)$$

Z corresponds to the coordinate in regard to equilibrium, Z' - bending, Z'' is proportional to the moment and Z''' - to the force. The shape of oscillations must meet the equation $Z^{IV} - \beta Z = \beta^4 A_d$. Usually the integration constants are found from the marginal side conditions, which are set to the rod ends. It is practical to introduce a new variable $u(x)=Z(x)+A_d$ and select the beginning of x coordinates at the loose end of the rod. Variable $u(x)$ expresses the difference of z coordinate between the fixed rod end and corresponding to x rod point. In this case, the integration constant A corresponds to the oscillation amplitude of the rod's loose end. The marginal conditions at the rod's fixed end will be $u(L)=A_d$, $u'(L)=0$ and at the loose end the marginal conditions are $u''(0)=0$, $u'''(0)=0$. It is obvious, that $C=D=0$, and $u(x)=AS_1(\beta x)+BS_2(\beta x)$. A is found by solving the equation system

$$\begin{cases} AS_1(\beta L) + BS_2(\beta L) = A_d \\ \beta\left[AS_4(\beta L) + BS_1(\beta L)\right] = 0 \end{cases} \quad (6)$$

In this case the rod's loose end oscillation amplitude is found

$$Z(0) = A = A_d\left(\frac{cosh\,\alpha + cos\,\alpha}{1 + cosh\,\alpha\, cos\,\alpha} - 1\right) \quad (7)$$

where $\alpha=\beta L$,

$$\beta = \sqrt[4]{\frac{\rho S\omega^2 - i\gamma\omega}{EI}} \quad (8)$$

The expression (8) becomes significant, when the denominator of the fraction is not equal to zero. When $\gamma=0$, denominator equality to zero expresses the oscillation resonance condition. The equation has got an infinite number of solutions, corresponding to bending oscillation modes with respective wave numbers β. $\alpha=\beta L$ roots are found using numerical techniques. Considering α values, the resonance mode frequencies are calculated using the equation

$$\omega_{0i} = \alpha^2 \frac{b}{L^2}\sqrt{\frac{E}{12\rho}}\,. \quad (9)$$

3. The investigation methodology

The micromechanical AFM sensor can operate as miniature cantilever microweight scales, when static cantilever bending, or cantilever dynamic parameter (oscillation frequency, phase, amplitude) changes are used for reading. Covering one plane of the micro cantilever with the gas-sensitive layer, the gas molecule adsorption in the cantilever causes strains [3], which are expressed by static microcantilever bending radius

$$\Delta\sigma = Eb^2/\left[4R(1-\nu)\right] \quad (10)$$

where $\Delta\sigma$ is microcantilever strains changes in "static mode"; E is Young module; b is microcantilever thickness; R is microcantilever bending radius; ν is Puasson coefficient.

The "static mode' method does not provide accurate enough information about the quantity of molecules adsorbed, as the strains in the sensor are formed only by the first layers of immobilized molecules.

In the realization of the micromechanical gas sensor method the adsorbed molecules of the gas sensitive layer change the oscillating system resonance frequency [4] and this change is expressed as

$$\Delta m = (k/4\pi)\,(f_1^{-2} - f_0^{-2}) \quad (11)$$

where Δm is microcantilever mass change due to adsorption; k is microcantilever construction coefficient, evaluating the material longitudinal toughness, microcantilever thickness b; width a; length L ($k = Eb^3w/4L^3$); f_0, f_1 are respectively, micro cantilever resonance frequencies before and after mass change.

Actually, a contact free micro cantilever can not only bend, but oscillates according other swinging degrees of freedom, so that the resonance frequencies (Fig. 2) of two-layer microcantilevers, used in the investigation, (Fig. 3) are found, applying finite element method (FEM) of the software package Ansys7.0.

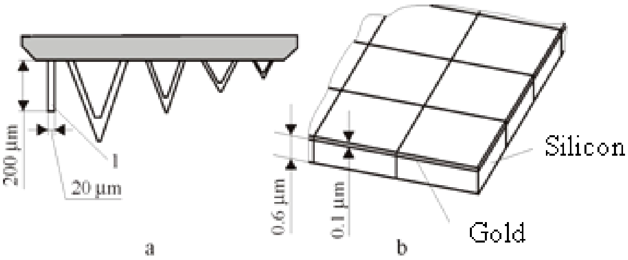

Fig. 2 The "Ultralever" scheme of AFM micro cantilever unit with the tested micro cantilever *1* (a) and the fragment of a two-layer micro cantilever FEM model (b)

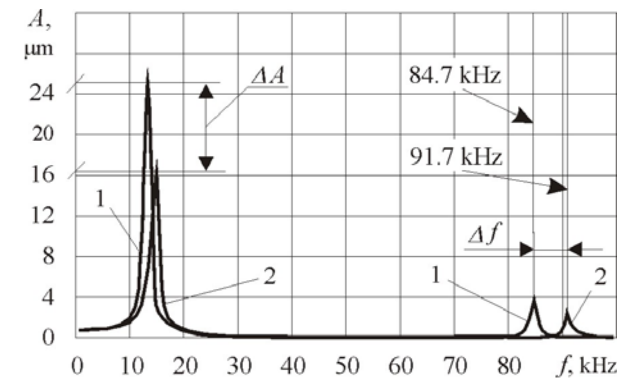

Fig. 3 Dependence of the microcantilever oscillation dynamic characteristics on the gas-sensitive layer density ρ: *1* - ρ = 19300 kg/m^3; *2* - ρ = 14475 kg/m^3; ΔA, Δf - respectively, the differences of oscillation amplitudes A_d and frequencies f in separate microcantilever oscillation modes

4. Investigation equipment and working modes

The operation principle of the micromechanical gas sensor is based on the response contact free AFM microcantilever oscillation parameters' to the detected gas.

The effect of environment chemical composition on the sensor dynamic characteristics was investigated after localizing AFM microcantilever in the original form of calibrated capacity (V=30 cm^3) medium – measurement chamber (Fig. 4). The detected gas composition in the chamber was formed using standard medical batchers, which enabled to regulate gas/air ratio within the range of 1%.

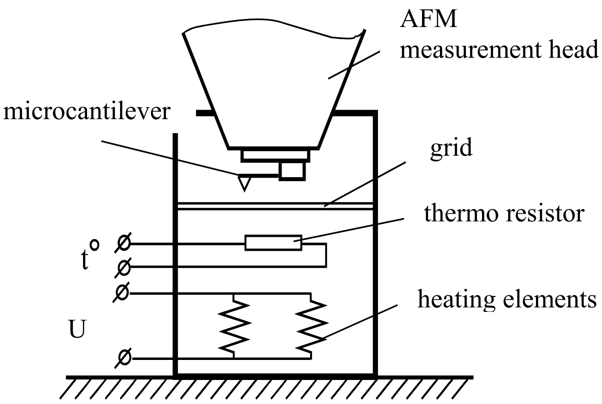

Fig. 4 The structural scheme of the measurement chamber

In order to achieve maximum sensitivity of the microcantilever in dynamic working mode, high frequency and minimal thickness microcantilevers (Figs. 2, 3) were selected, the plane of one was covered with gas-sensitive material SnO_2, In_2O_3, Al or Au (in the study experimental dependences were set, using gold covered microcantilevers).

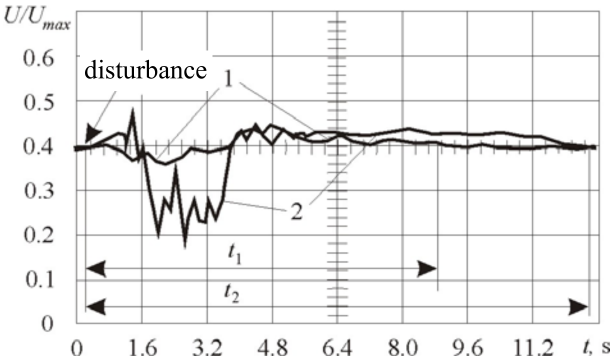

Fig. 5 The effect of 0.4 m/s speed pulse aerodynamic disturbance on different AFM systems measurement methods (the microcantilever is contact free with the surface): *1* – realizing the phase changing method; *2* – applying the amplitude measurement method. U/U_{max} - AFM scanner control voltage in relative units; t - time; t_1, t_2 - respectively, system steady-state times, realizing phase changing and amplitude measurement methods

After the evaluation of microcantilever dynamical working mode the effect of FEM modeling results and test performance of 0.4 m/s speed pulse aerodynamic disturbance (probable during the experiment) on micro cantilever sensitivity, the measurement systems and additional equipment working modes were set, which provided the maximum value of signal/disturbance ratio:

- the micro cantilever is excited by resonance mode (f=84.7kHz), where significantly higher useful signal level in comparison to basic resonance frequency is ensured (Fig. 3);
- the measurements are made applying the phase changing method, which is approximately 4 times less sensitive to the environment turbulence than the amplitude method (Fig. 5);
- the measurement results are registered in the „Force- Distance" working mode, when the microcantilever is contact free with the surface.

5. Investigation results

During the course of investigation the detected environment formation peculiarities and the effect of source indefiniteness on measurements have been determined; the micromechanical gas sensor responses to saturated water vapor, CO_2 and CO gas environments and the sensor's response to C_2H_5OH and mercury vapor in the air have been quantified.

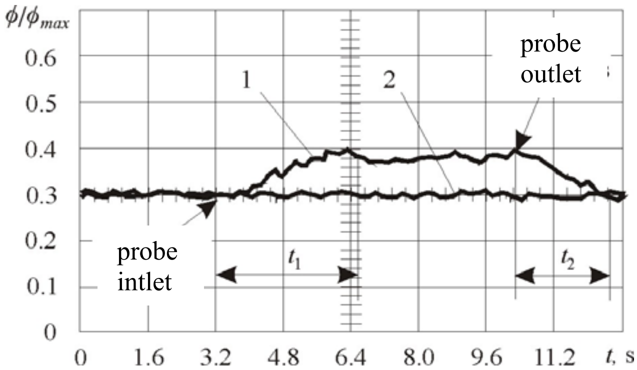

Fig. 6 Evaluation of microcantilever sensitivity to mercury vapor: *1* – Hg concentration characteristics; *2* – telltale characteristics without Hg vapor; t_1 – steady-state (adsorption) time; t_2–sensor regeneration time; ϕ/ϕ_{max} - sensor excitation and outgoing signal change in relative units; t - time

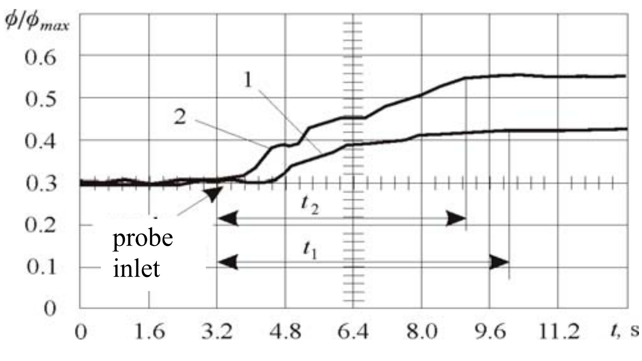

Fig. 7 Dependence of microcantilever output signal change in time t on CO_2 concentration: *1*- 60% concentration; *2* – 80% concentration; t_1, t_2 - measurement system steady-state times at appropriate concentrations; ϕ /ϕ_{max} - sensor signal change in relative units; t - time

In the study experimental dependences were set using gold covered microcantilever (Fig. 2) and they illustrated the selection of the system measurement method (Fig. 3), measurement system response to aero dynamical disturbance at different working modes (Fig. 5), system dynamic characteristics (steady-state, regeneration times), the micro cantilever sensitivity to different gas concentrations (Fig. 6, 7).

Fig. 6 presents the investigation results of mercury vapor effect on the outgoing signal. The mercury vapor was formed when a mercury drop was kept at room temperature for 24 hours in a hermetic 2 cm^3 capacity vessel.

The sensor response to different CO2 concentrations quantitative aspects are presented in Fig. 7.

6. Conclusions

1. Mathematical model of a microcantilever gas sensor is made.
2. Microcantilever gas sensor dynamical working mode characteristics, applying the finite element method, are set.
3. On the basis of AFM complex a measurement system (equipment and methodology) for the evaluation of gas microsensors response is worked up.
4. Initial investigations of Au, Al, SnO_2 and In_2O_3 coating sensitivity of microcantilever gas sensors, working at low temperatures, in the environment with CO_2, CO, Hg, H_2O and C_2H_5OH components, are made.
5. Setting of individual sensor coating response to different gases, gives the opportunity to use microcantilevers, covered with selectively adsorbing different gas coatings, in electronic odor identification equipment – "electronic noses".

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MIKROMECHANINIO DUJŲ JUTIKLIO JAUTRIO TYRIMAS

R e z i u m ė

Straipsnyje pateikti atominių jėgų mikroskopo (AJM) matavimo mikrogembės dinamikos tyrimo rezultatai, gauti AJM sistemoje įdiegiant aplinkos dujinei sudėčiai jautrių „kvarcinių mikrosvarstyklių“ režimą.

AJM mikrogembės dinamika dujų jutiklio darbo režime išreikšta matematiškai, naudojant paskirstytųjų parametrų modelį baigtinių elementų metodu nustatyti mikrogembės rezonansiniai dažniai bei jų priklausomybė nuo jos struktūros pokyčių. Eksperimentiškai ištirta aplinkos cheminės sudėties įtaka mikrogembių, padengtų dujoms jautriu sluoksniu, dinaminėms charakteristikoms.

Tyrimo rezultatai naudotini kuriant selektyvių mikromechaninių dujų jutiklių sistemas – „elektronines nosis“.

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INVESTIGATION OF MICROMECHANICAL GAS SENSOR RESPONSE

S u m m a r y

The results of microcantilever dynamics investigation, atomic force microscope (AFM) system in realization of the regime of "quartz balance" are given in the article.

The dynamics of AFM microcantilever in gas sensor regime is expressed mathematically. Using the devoted elements model, AFM microcantilever resonance modes, their dependence upon the changes of microcantilever structure are determined. Experiments are done with the influence of chemical content of the environment on dynamic characteristics of the microcantilever, covered by sensitive to gas film.

Research results are used in creating selective microcantilever gas sensors systems – electronic nose (e-nose).

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

ИССЛЕДОВАНИЕ ЧУВСТВИТЕЛЬНОСТИ МИКРОМЕХАНИЧЕСКИХ ДАТЧИКОВ ГАЗА

Р е з ю м е

В статье представлены результаты исследования динамики измерительной микроконсоли микроскопа атомных сил (МАС), полученные реализуя в системе МАС режим работы "кварцевых микровесов", чувствительных к химическому составу окружающей среды.

Динамика микроконсоли МАС в режиме датчика газа описана математически, используя модель распределенных параметров. Методом конечных элементов определены частоты резонанса и их зависимость от изменений структуры микроконсоли. Экспериментально исследовано влияние химического состава среды на динамику микроконсоли с покрытием, обладающим селективной чувствительностью к составу газа.

Результаты исследования могут быть использованы при создании системы микромеханических датчиков газа - "электронного носа".

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