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Adsorption–desorption noise in QCM gas sensors

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ABSTRACT

There are several mechanisms that are related to the fluctuation phenomena in QCM. The aim of our research is to study the sensitivity and the influence of different kinds of noise on sensor resolution. Our experiments are performed on sensors with a sorption layer of polypyrrole which is suitable for detection of water vapor. Based on these experiments, we conclude that $1/f$ noise caused by quartz internal friction and adoption–desorption (generation–recombination) noise from analyzed gas cause the main components of the measured noise spectral density. The adoption–desorption noise power depends on the physical and chemical parameters of the analyzed gas and it is proportional to the gas density. The adsorption–desorption kinetics is described by Kolmogorov's equation and is compared with the Wolkenstein and Langmuir equations.

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1. Introduction

Quartz crystal microbalance (QCM) represents a high-sensitivity sensor for detection of chemical substances, and is widely utilized as a result of its robust nature, availability and affordable interface electronics [1]. The heart of the sensor is AT-cut quartz crystal whose electrodes are covered by sorption layers with affinity to the molecules of the detected matter. Since the resonance frequency of quartz crystal depends on the total oscillating mass, the principle of QCM is a frequency shift caused by the addition of sorbed matter (molecules of the detected mass) to the electrodes or its removal from them. Thus, the sensitivity depends on the stability of the oscillator and the accuracy and stability of the devices measuring the parameters of the quartz resonator. The selectivity is given by the choice of the material of the sorption layer.

Analyses of noise measurements represent the approach of extracting more selective response from chemical sensors, such as resistive [2–6] and surface acoustic wave [7] sensors. Experimental results showed that the noise spectral density of the sensor's resistance fluctuations is modified by exposure to different gases as well as by exposure to different concentration of gases [5]. Thus, noise spectroscopy might be highly useful for improving gas sensors selectivity if both theoretical models and adequate

sensing devices can be developed [3]. Gomri et al. [4] proposed a model of adsorption–desorption noise in metal oxide gas sensors, based on the free electron density fluctuation produced by the gas adsorption. Using this model for simulating the oxygen chemisorption-induced noise, they found that the contribution of oxygen adsorption–desorption noise to the noise spectra is a Lorentzian component having a corner frequency and low frequency magnitude which are specifics of the adsorbed gas.

The paper deals with an experimental study how absorption of detected gas molecules affects frequency fluctuations of QCM, and presents the model of adsorption–desorption noise which is devoted on the basis of Kolmogorov equation for interaction between two reservoirs.

2. Experimental setup

Two standard approaches of QCM measurement exist, namely the active method and the passive method. In the first one, the quartz crystal is a part of a wideband oscillator circuit whose frequency is controlled by the crystal properties [1] and is measured by a frequency counter with a resolution of 1 Hz, 0.1 Hz or 0.01 Hz. Some researchers [6] use a reference oscillator to avoid ambient effects. In that case, a counter measures the frequency difference between the output signal of the sensor based oscillator, and the output signal of the reference quartz based oscillator. In the passive approach, a QCM receives a frequency which is determined by an external source [2]. Thus, the frequency shift and the shift in bandwidth (proportional to dissipation) can be obtained by recording

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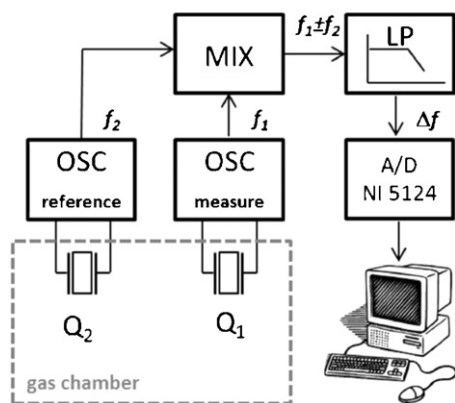


Fig. 1. Noise measurement setup for quartz crystal microbalance.

the complex admittance around a resonance frequency and fitting resonance curves to the admittance spectra.

In order to study a small frequency fluctuation of QCM, the active method was modified to measure instantaneous frequency of QCM. The scheme of our measurement setup is shown in Fig. 1. The measurement setup consists of a quartz crystal with deposited sorption layers (measuring oscillator) and a quartz crystal without sorption layers (reference oscillator). These crystals are driven by two independent oscillator circuits that regulate frequency of each quartz crystal at the minimum impedance which corresponds to serial resonance. The frequency difference Δf between these two oscillators is a result of mixer procedure and low-pass filtering. This signal is led to an input of a data-acquisition card (NI 5124) which triggers on the rise-edges of the output signal and stores the corresponding times at which the instantaneous frequency is estimated by own-written software.

3. Samples preparation

The experiments were performed on sensors with a thin sorption layer of polypyrrole (PPY) which is a material suitable for construction of QCM humidity sensors. The change of relative humidity from 0% RH to 25% RH causes a change of resonant frequency (Δf) in the range 50–150 Hz. Sensor parameters (e.g. sensitivity, dynamic properties, time stability of response) depend not only on the composition but even on the way of deposition of the sorption layer. Thus, PPY was deposited using Matrix Assisted Pulsed Laser Evaporation (MAPLE). This technology [12–16] is a laser deposition method providing a gentle mechanism for organic layer deposition. In MAPLE, a frozen phase consisting of a dilute solution of a high-molecular weight compound (basic material) in a low-molecular-weight solvent (matrix) is used as the laser target. Deposition takes place after the impact of a laser pulse with the surface of the frozen target. In an optimal case, the energy of the laser pulse is completely absorbed by the matrix, resulting in a strong local increase of temperature. The matrix molecules transfer their kinetic energy of thermal motion to the molecules of basic material. Hence the molecules of basic material are transferred to the substrate “mechanically”, with neither photolytic nor pyrolytic damage.

The advantages of MAPLE in comparison with conventional methods can be summarized as follows: (a) the thickness of the prepared layers can be simply controlled by setting laser fluence and the number of pulses. Thus, reproducible preparation of thin layers having thicknesses of the order of tens of nanometers is possible. (b) The prepared layers are porous (i.e. they have a high surface/volume ratio), so they are suitable for sensor applications. (c) “Sandwich” structures can be fabricated in situ in one step by

changing targets during the deposition. (d) The effect of the laser pulse is strongly limited in time and localized in space, hence there is a possibility to create various patterns by Matrix-Assisted Pulsed Laser Evaporation – Direct Write method.

The source target for MAPLE was prepared from 5 wt.% water solution of polypyrrole (Mw = 10,000) doped with dodecyl sulfonic acid-specific electrical conductivity of the solution: $\sigma = 10\text{--}40 \text{ S cm}^{-1}$ (Sigma Aldrich). This solution was stirred and homogenized by an ultrasonic device. Subsequently, it was frozen to -196°C in the tubular mould using liquid nitrogen. The prepared target was inserted into the deposition chamber and placed on the rotating shaft of the target holder. The holder was simultaneously cooled by liquid nitrogen. Uniform ablation of the material from the target surface was achieved by constant rotation during the deposition process. The deposition chamber was evacuated by a turbomolecular pump to a residual pressure of $5 \times 10^{-3} \text{ Pa}$. The pressure in the chamber did not exceed 3 Pa during the deposition process (a background gas was not used).

The depositions were carried out using Nd:YAG laser (Quantel), operating at the fourth harmonic frequency (wavelength 266 nm). The Nd:YAG laser was used in a pulse mode with a 10 Hz repetition rate. The fluence F of the laser radiation was set by an attenuator to be $F = 0.4 \text{ J cm}^{-2}$. The substrate-quartz crystal (without any surface masking) was placed at a distance of 60 mm. The deposited PPY layers have a thicknesses ranging from 80 to 400 nm.

Fig. 2 shows good agreement between relative humidity measurements, where a commercial available humidity sensor (FH A646-1, ALBHORN) and a QCM with deposited polypyrrole were used. RH increase from 25% to 85% causes a rise of frequency difference Δf between measuring oscillator and reference oscillator for about 400 Hz.

4. Results and fluctuation mechanisms in QCM

Several mechanisms related to fluctuation phenomena exist in QCM. The fundamental noise includes thermal noise, $1/f$ noise due to the quantum fluctuation and phonon scattering by defects, adsorption–desorption noise from the analyzed gas, thermo-mechanical noise, temperature fluctuation noise, and electronic oscillator noise. The mechanisms can be separated into two groups; the first one is related to the quartz crystal and its electrical circuit, while the second one is related to the adsorption–desorption processes on a sorbent layer. Experimental

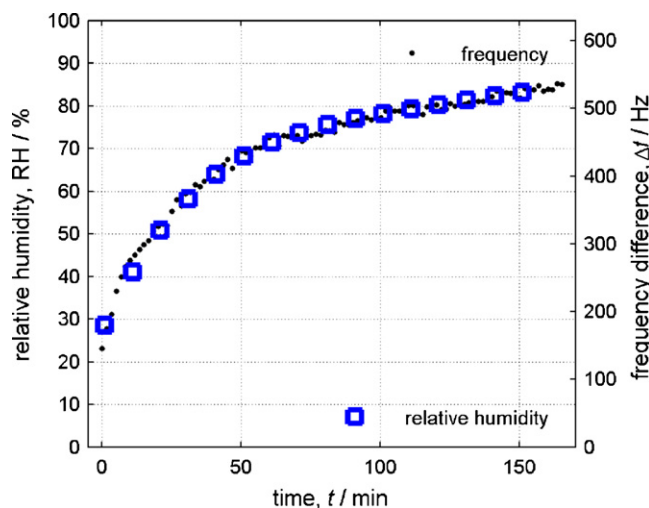


Fig. 2. The frequency difference Δf between measuring and reference oscillators, and relative humidity as a function of time after certain amount of water being put into gas chamber and sensors being exposed.

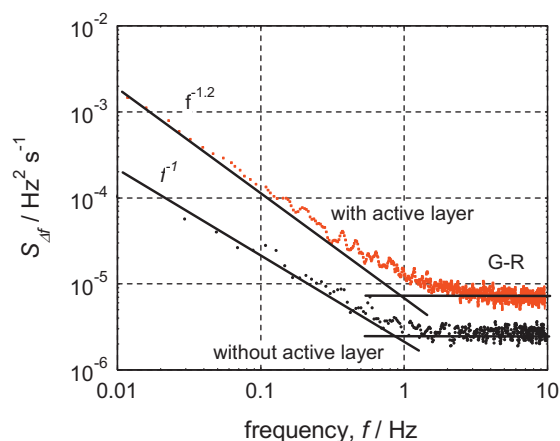


Fig. 3. The spectral density $S(\Delta f)$ of frequency difference fluctuation of the polypyrrole QCM at relative humidity 30% and spectral density of frequency difference fluctuation of the two identical quartz crystals without active layer.

results shown in Fig. 3 reveal that the $1/f$ noise, thermal noise and generation–recombination (adsorption–desorption) noise seem to be the main noise components. Fig. 3 also shows the spectral density of QCM has a higher slope of $1/f$ noise part and a higher value of GR component in comparison to the noise spectrum of the measurement system, which was determined on the basis of the measurement of frequency difference fluctuation using two identical quartz crystals without active layer.

There are various models and mechanisms that could be responsible for the observed noise, especially for $1/f$ noise (e.g. [8–11]); however, this paper focuses only on fluctuation mechanisms connected with chemical processes on the active layer of QCM.

Fig. 4 illustrates how the spectral density $S_{\Delta f}(f)$ of the frequency difference fluctuations is affected by the absorption/desorption of water molecules by the QCM sensor. When the RH increases, the $1/f$ noise component changes insignificantly while the G–R component noticeably increases with a rise of RH value. The authors of this paper suppose that thermal noise component and $1/f$ noise component are mainly associated with quartz crystal properties represented by its equivalent circuit, while generation–recombination noise results from adsorption–desorption processes which are present on the active layer of the QCM sensor. Further, it can be assumed that a shift of G–R noise is caused by increased flux density between the sorbent layer of QCM and the ambient environment. Considering these interactions between the two reservoirs, a model of

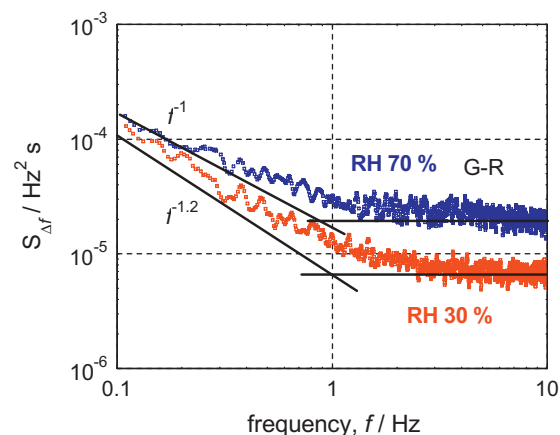


Fig. 4. Spectral density $S(\Delta f)$ of frequency difference fluctuations of the polypyrrole QCM at relative humidity 30% and 70%.

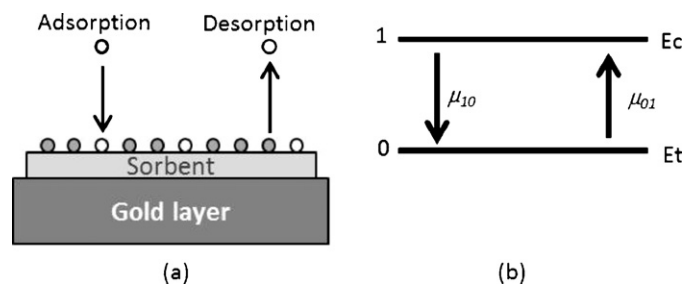


Fig. 5. (a) a model of adsorption–desorption processes on the quartz crystal layer and (b) the transition probability intensity for emission and capture.

adsorption–desorption noise could be formulated on the basis of Kolmogorov equation.

5. Adsorption–desorption noise

This section presents a model of adsorption desorption noise related to RH concentration. The value of the adoption–desorption noise depends on the physical and chemical parameters of the analyzed gas. Adsorption–desorption kinetics is described by the Kolmogorov equation and compared with the Langmuir and Wolkenstein equations.

5.1. Kolmogorov equation

A proposed model of adsorption–desorption noise is based on interaction between two reservoirs:

- analyzed gas with concentration n , temperature T and partial pressure p ,
- sensor sorbent surface with total surface density of sites for the adsorption of analyzed gas N_0 , with surface density of sites occupied by analyzed gas molecules N_t

5.1.1. Transition probability intensities

The model supposes that the system is: (i) Markovian, (ii) near equilibrium, and (iii) generation–recombination processes may take place between these two reservoirs. The random process of the surface site is assumed to have two states and to be stationary with a constant transition probability density μ_{ij} defined by

$$\mu_{ij} = \lim_{t \rightarrow s} \frac{p_{ij}(s, t)}{t - s} \quad (1)$$

where p_{ij} devotes the transition probability at time s from the i -state to the j -state at time t . Fig. 5a shows the model of adsorption–desorption process on the quartz crystal sorbent layer. Desorption corresponds to molecule emission from sorbent, i.e. generation of free particle, and the process of adsorption corresponds to recombination. Thus, the process is similar to generation–recombination in semiconductors. The transition probability density for emission and capture is schematically shown in Fig. 5b, where μ_{01} is the transition probability density for the molecule emission and μ_{10} for capture. There are two physical quantities: the characteristic time τ_c for a molecule to be captured on the surface site which is inversely proportional to the transition probability density μ_{10}

$$\mu_{10} = \frac{1}{\tau_c} \quad (2)$$

and the characteristic time τ_e for molecule emission from the surface site which is inversely proportional to the transition probability density μ_{01}

$$\mu_{01} = \frac{1}{\tau_e} \quad (3)$$

The probabilities $p_{ij}(t)$ of the transition from the state i into the state j are found by solving the Kolmogorov differential equations

$$\frac{dp_{ij}(t)}{dt} = \sum_{k \in I} \mu_{ik} p_{kj}(t) \quad (4)$$

for $i, j = 0, 1$, with the conditions $p_{ii}(0) = 1, p_{ij}(0) = 0$, for $i \neq j$. In thermodynamic equilibrium, the statistics, that the surface site is free or occupied by a molecule, is described by the absolute probability distributions Π_0 and Π_1 . These ones are given by solving the Kolmogorov equation [17] for stationary state in the form

$$\sum_{k \in I} \Pi_i \mu_{ik} = 0, \quad (5)$$

where

$$\Pi_0 = \left[1 + \frac{\mu_{01}}{\mu_{10}} \right]^{-1} \quad (6)$$

$$\Pi_1 = \left[1 + \frac{\mu_{10}}{\mu_{01}} \right]^{-1} \quad (7)$$

The absolute probability distributions Π_0 and Π_1 are similar to the Fermi–Dirac statistics.

5.1.2. Kinetic equation

Kinetic equation describing surface density of adsorbed molecules follows from (5) in the form

$$\frac{dp_{10}}{dt} = \mu_{10} \Pi_0 + \mu_{11} \Pi_1 \quad (8)$$

$$\frac{dp_{01}}{dt} = \mu_{00} \Pi_0 + \mu_{01} \Pi_1 \quad (9)$$

where μ_{00}, μ_{11} are densities that the system is persisting in the state 0 or 1, respectively. With $\mu_{01} = C_N n_1$ for generation, $\mu_{10} = C_N n$ for recombination and C_N as coefficient of capture,

$$\frac{dN}{dt} = \text{adsorption}(N) - \text{desorption}(N) = C_N n(N_0 - N) - C_N n_1 N. \quad (10)$$

The surface density of adsorbed molecules is proportional to the coefficient of capture C_N , concentration of adsorbed molecules n , sensor surface density of sites N_0 and the effective concentration of occupied sites n_1 .

5.2. Langmuir adsorption kinetics

The rate of formation of the monolayers can be written as [18]

$$\frac{d\Theta}{dt} = \text{adsorption}(\Theta) - \text{desorption}(\Theta) = k_a(1 - \Theta)C - k_d\Theta \quad (11)$$

where $\Theta = N/N_0$, C denotes concentration of analyzed gas molecules, k_a and k_d are the rate constants for adsorption and desorption processes, respectively. Integration of (11) and substitutions $k_{obs} = k_a + k_b$ and $k' = C/(C + k_d/k_a)$ lead to

$$\Theta(t) = k'(1 - e^{-k_{obs}t}) \quad (12)$$

If the fractional coverage Θ is measured as a function of time t , coefficients k_{obs} and k' can be determined by fitting of (11) on the experimental data.

5.3. Wolkenstein adsorption kinetics

The rate of formation of monolayers is [3,4]

$$\begin{aligned} \frac{dN}{dt} &= \text{adsorption}(N) - \text{desorption}(N) \\ &= \alpha p(N_0 - N) - N\nu \exp\left(\frac{-E_b}{kT}\right) \end{aligned} \quad (13)$$

$$\alpha = \frac{\Pi\sigma}{\sqrt{2\pi mkT}} \quad (14)$$

$$p = nkT \quad (15)$$

where Π is the probability that a molecule approaching an adsorption center will be fixed on the sensor surface, p is the pressure of adsorbed gas, m is the adsorbed molecule mass, σ is the sensor site adsorption cross section, E_b is the binding energy of the adsorbed molecule and ν is the oscillation frequency of the corresponding particle (typically $\nu = 10^{12}$ Hz).

5.4. Modeling of the adsorption–desorption noise

In order to find the dependence of the fluctuation $\delta N(t)$ around the value N_0 at the adsorption–desorption equilibrium on the thickness of the sensing layer, small fluctuations of N around the value N_0 are considered as done in [3,4]. After some calculation procedures, the following differential equation is obtained [4]:

$$\frac{d\delta N}{dt} = -\frac{\delta N}{\tau} \quad (16)$$

where

$$\tau = \left[\left(\frac{d(\text{desorption}(N))}{dN} - \frac{d(\text{adsorption}(N))}{dN} \right)_{N_0} \right]^{-1} \quad (17)$$

In order to determine the power spectral density of the adsorbed molecules density fluctuation, Wiener–Khinchin theorem [19] can be applied. The theorem states that spectral density of a wide-sense stationary random process (domain $\omega \in (-\infty, \infty)$ is considered) is the Fourier transform of the corresponding autocorrelation function. Since frequencies can be only positive in physical processes, the above mentioned spectral density equals two times Fourier transform. Autocorrelation function is an even function, thus, factor two is included again, the power density spectrum of the adsorbed molecules density fluctuation writes [20]:

$$S_{\delta N} = 4 \int_0^{+\infty} \sigma^2 e^{-|t|/\tau} \cos \omega t dt = \frac{4\sigma^2 \tau}{1 + \omega^2 \tau^2}, \quad (18)$$

where σ^2 denotes the mean square value of δN .

As mentioned above, the experimental results of humidity measurements show that the concentration of detected matter affects the spectral density of frequency difference fluctuations. The low-frequency component ($1/f$ noise) changes insignificantly, while G–R component increases with a rise of concentration. Thus, it can be assumed that a shift of G–R noise is caused by increased flux density between the sorbent layer of QCM and the ambient environment. The increased flux of molecules corresponds to the increased variation of frequency difference fluctuations. Relation (18) can be rewritten

$$S_{\Delta f} = \frac{4\sigma_{\Delta f}^2 \tau}{1 + \omega^2 \tau^2}, \quad (19)$$

where $\sigma_{\Delta f}^2$ denotes the variance of frequency difference fluctuations. On the basis of the model and experimental results (frequency change and its second statistical moment), the time τ for molecule capture can be estimated. Concerning Fig. 4 and the

relative humidity 30% at frequency 2 Hz, the time constant equals to 1.26 μ s and is calculated on the basis of parameters estimated from the measurement, spectral density $S_{\Delta f} = 7.26 \times 10^{-6}$ Hz²/s and standard deviation $\sigma_{\Delta f} = 1.21$ Hz. For the relative humidity 70% at frequency 2 Hz, the time constant $\tau = 1.23$ μ s is calculated from values $S_{\Delta f} = 2.15 \times 10^{-5}$ Hz²/s and standard deviation $\sigma_{\Delta f} = 2.11$ Hz.

6. Conclusions

Experimental results showed that absorption of detected matter affects the frequency fluctuations. The paper focuses on adsorption–desorption noise without consideration of the diffusion process in an active layer, and presents its model, which is developed on the basis of the Kolmogorov equation for interaction between two reservoirs. The probability, that molecules will be captured on an active layer of the sensor, is proportional to the adsorbed molecule thermal velocity and the surface site cross section. The probabilities, that surface sites are free or occupied by molecules, are implied by the Kolmogorov equation in the case of thermodynamic equilibrium, when the flux of emitted particles is the same as the flux of captured particles on the active layer of the sensor. The derived probability distributions are similar to the Fermi–Dirac statistics for semiconductors. The surface density of adsorbed molecules is proportional to the surface site cross section, adsorbed molecule thermal velocity, concentration of adsorbed molecules and sensor surface density of the sites. The adsorption–desorption kinetics described by the Kolmogorov equation is compared with the Wolkenstein and Langmuir equations.

The observed relative changes of noise spectral density at different RH values correspond to the frequency shift. Further, these changes represent information, which can enhance selectivity and sensitivity of the QCM sensor. On the basis of the model and experimental results (frequency change and its second statistical moment), the time τ for molecule capture can be estimated which can give additional information about processes on the sorption layers. Thus, we can conclude that fluctuation enhanced noise sensing can be utilized for gas measurements by QCM sensors.

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