Critical Consideration In Biomaterıal Surfaces: A New Approach to Thin Film Thickness Measurements

Mebrure, M., Erdogan*
Biomedical Engineering, Faculty of Engineering, Baskent University, Ankara, Turkey
mbrrerdogan@gmail.com

Kadir, K.C., Erbas
Biomedical Engineering, Faculty of Engineering, Baskent University, Ankara, Turkey
kcerbas@gmail.com

Ismail, I.C., Kocum
Biomedical Engineering, Faculty of Engineering, Baskent University, Ankara, Turkey
kocum@baskent.edu.tr

Dilek, D., Cokeliler Serdaroglu
Biomedical Engineering, Faculty of Engineering, Baskent University, Ankara, Turkey
cokelilerdilek@gmail.com

ABSTRACT

Thin film coating is used to provide various functions to surfaces and therefore it is a technology that contributes significantly to the country's economy. With coatings at the thin film level, the quantum properties of materials are revealed, which plays a crucial role in the development of materials with new and unique properties. In the field of biomaterials, nano-functionalized surfaces obtained with thin film coatings have promising biological properties. Since the thin film coating measurements used so far cannot make precise measurements and are very costly, new methodologies should be introduced for thin film thickness measurements. We present a portable, low-cost, mass-sensitive QTF (Quartz Tuning Fork) sensor system capable of frequency measurements. In recent years, QTFs have begun to be applied as transducers to sensor systems with the potential to produce highly reliable and low-cost mass-sensitive sensors. Additionally, QTFs are known as common oscillator components because of their stable resonant frequencies. In this study, mass-dependent frequency values deposited on the QTF transducer surface with plasma coating were measured using an advanced QTF mass-sensitive sensor system. According to the frequency shift values, mathematical approaches have been introduced to the thin film thickness formed on the QTF surface and future perspectives are presented.

CCS CONCEPTS

• Hardware; **• Hardware test**; **• Analog, mixed-signal and radio frequency test**;

KEYWORDS

Quartz tuning fork, Thin film coating, Plasma coating, Mathematical equations, Biomaterial

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1 INTRODUCTION

As is known, thin film coating is used in almost all kinds of industries to give surfaces various functions. Today, thin film coating is successfully applied in superconductor technology, military technologies, aerospace, electronic semiconductor devices, optical coatings, energy generation and storage, and healthcare (e.g., medical implants). For this reason, thin-film coating applications are a technology that contributes significantly to the national economy [1]. Quantum-level properties of materials emerge at the thin-film level and as the thin-film thickness changes, the structural, optical, and electrical properties of the materials change. In some applications, the coating thickness is so thin and its control is critical that, for example; with the coatings made at the level of thickness equivalent to the wavelength, the construction of selectively permeable or selectively suppressing optical filters can be achieved.

In the field of biomaterials, however, the material surface plays an important role in adapting the in-vivo response by controlling the body's biological response. Therefore, the surface of biomaterials has become one of the most intense research topics. A nano-functionalized surface has promising biological properties, and clinical applications of biomaterials can be enhanced by fabricating a nanostructured surface. For this reason, thin film coating studies are carried out to improve biocompatibility, blood compatibility, biodegradability, corrosion resistance, and mechanical and antibacterial properties of biomaterials used in the human body [2], [3], [4], [5], [6]. Consequently, thin-film coating thickness plays an important role in the development of materials with new and unique properties.

The methods used to measure thin film thickness can be grouped into spectroscopic, microscopic, and quartz-based methods. However, spectroscopic and microscopic methods have disadvantages such as not being able to measure sensitively, not being able to control the accumulation rate instantaneously, requiring high-cost equipment, and requiring highly specialized personnel [7]. For these reasons, new approaches to thin film thickness measurements are required to overcome such disadvantages.

The technique we recommend for thin film thickness measurements is the QTF (Quartz Tuning Fork) transducer, which is a mass-sensitive quartz-based technique. QTF is a quartz-based, self-exciting, and self-sensing resonator with a fork-shaped mechanical structure. Similar to tuning forks used in music, QTFs can vibrate at their resonant frequency with a driving force – an electronic signal. If mass is added to the quartz crystal, a shift occurs in the resonant frequency and this shift is proportional to the mass. QTFs have widespread use in electronics because they are cheap, small in size, and suitable for mass production, as well as vibrating at a constant frequency for a long time, and they first entered the literature with Forrer's study published in 1969 [8]. The quartz crystal monitor offers several advantages over other thin-film thickness measurement methods. Because it is only mass-sensitive, it can be used to monitor the deposition of any evaporating material, metal, semiconductor, or insulator. It also combines the high precision of the quartz crystal with the precision of frequency measurements. Therefore, measurement of extremely thin films or low deposition rates can be achieved relatively easily. Finally, frequency signals can be converted to analog control signals to obtain deposition rates and control evaporation [9]. QCM (Quartz Crystal Microbalance), which is a mass-sensitive quartz-based method, is the most widely used and closest technique to the technique we have presented in thin film thickness measurements However, QCM can be highly affected by ambient forces due to its geometry and low-quality factor. In addition, QCM monitors have disadvantages such as shifting of oscillator frequencies, non-linearity in pulse-analog frequency counter, short sensor life (although periodic sampling techniques can expand the range of the crystal), common limitations in physical vapor deposition techniques, sensitivity due to stress changes, requiring geometric and adhesion coefficient calibration [8]-[10]. Quartz tuning fork (QTF) has proven to be suitable for the high-quality factor, sharp frequency response, high sensitivity, long stability, and mass detection compared to the most widely used QCM for thin film thickness measurements. In addition, its low cost compared to other thin film thickness measurement methods is another advantage [11].

In this study, mass deposition was achieved on the QTF surface by coating the QTF sensors in a low-pressure plasma environment. Low-pressure plasma processes provide a wide range of technical applications and modification strategies in the field of coating technology. The plasma technique, due to its high flexibility and versatility, facilitates a wide range of surface adjustments, for example in polymer processing, medical applications, or textiles. Therefore, a wide range of different gases or gas mixtures can be used to functionalize or coat various substrates [12]. The frequency shift created by the mass accumulation on the QTF was read through the QTF mass-sensitive sensor system. This study aims to determine the thin film thickness value depending on the amount of mass accumulated by the frequency shift of the QTF sensor and to bring mathematical approaches to the change in the resonance frequencies of the QTF sensors with the coated film.

2 MATERIAL AND METHOD

2.1 Materials

Quartz Tuning Fork transducers: GWX-26 was purchased from Golledge Electronics Ltd. These transducers came with a sealed cylindrical cap covering the Quartz Tuning Fork under vacuum. The dimensions of the cylindrical structure are 2.0 x 6.2mm. The resonant frequency of the forks is 32,768Hz. The plasma deposition experiments were performed with a Lab-Made (Radio Frequency) plasma device in the Baskent University Biomedical Engineering Biomaterials and Biosensor Applications laboratory. QTF mass-sensitive sensor system has been created as given in Unal 2016 [13]. The system consists of a high-speed DDS core AD9850 and a microcontroller board. A more detailed explanation can be found in Unal 2016.

2.2 Plasma coating experiment set-up

For plasma coating experiments, 11-MUDA (mercaptoundecanoic acid 95%) chemical and QTF transducers were used. 0.8480 grams of 11-MUDA chemical was prepared and poured onto the glass slide and two QTFs were used for this experiment. QTF surfaces were placed near the 11-MUDA chemical on the glass slide, facing the plasma radiation. The plasma power was set at 60 W and the pressure at 1.5 E^{-1} torr. Each 11-MUDA plasma deposition application was carried out for 2 minutes. The results of each frequency measurement made on the QTF sensor system were read on a laptop computer via a user-friendly interface (details can be found in [13]). Each measurement gives a result of the resonant frequency of the QTF that is at its peak, which is the highest amplitude at the newly shifted frequency. The frequency difference (Δf) can be calculated by subtracting the mass-loaded QTF frequency (f1) from the bare QTF frequency (f0=32,768 Hz).

2.3 Mathematical Equations

The frequency formula of a TF (Tuning Fork) is derived from the oscillation calculations of a cantilever beam. Prongs of the tuning forks vibrate in a symmetrical mode which corresponds to the first fundamental mode of a cantilever beam [14]. A tuning fork can be approximated to a cantilever vibration with an appropriate choice of effective length which is between the inner and outer edges of the prongs [15], [16]. This length can be determined to give the measured frequency value. Consequently, in this study, the frequency of the tuning fork is modeled based on the first fundamental mode of fixed beam oscillation at one end. Euler–Bernoulli beam theory explains the oscillation of a cantilevered beam as follows.

$$-EI\frac{\partial^4 y}{\partial x^4} = \mu \frac{\partial^2 y}{\partial t^2} \tag{1}$$

In Eq.1, E, I, and μ represent Young's Modulus, the second moment of area of the cross-section, and the linear mass density of the beam. Parameters x and y are coordinates of a point on the beam and t is time [17]. Solution of this PDE (Partial Differential Equation) with the boundary conditions given in ref. yields an angular frequency formula for the first fundamental mode as in Eq.2.

$$w = \frac{3.516}{L^2}\sqrt{\frac{EI}{\mu}} \tag{2}$$

In Eq.1, the term EI is named flexural rigidity and the term $EI\ \partial^2 y/\partial x^2$ gives the bending moment of the beam. The basic concept of our study is that when the beam is covered with a homogeneous film, the bending moments (*EI*) and linear densities (μ) are additive. Therefore, in a film-wrapped beam, the Euler-Bernoulli equation can be rewritten as follows.

$$-\left(E_1 I_1 + E_2 I_2\right) \frac{\partial^4 y}{\partial x^4} = \left(\mu_1 + \mu_2\right) \frac{\partial^2 y}{\partial t^2} \tag{3}$$

In this case, the angular frequency will be as in Eq.4 where the subscripts 1 and 2 denote the values of the beam and the film respectively, and w' represents the new frequency of the coated beam. (L: Length of cantilevered beam)

$$w' = \frac{3.516}{L^2}\sqrt{\frac{E_1 I_1 + E_2 I_2}{\mu_1 + \mu_2}} \tag{4}$$

Eq.4 can be rewritten so that the frequency of the beam is a coefficient as in Eq.5.

$$w' = \frac{3.516}{L^2}\sqrt{\frac{E_1 I_1 + E_2 I_2}{\mu_1 + \mu_2}} = \frac{3.516}{L^2}\sqrt{\frac{E_1 I_1}{\mu_1}}\sqrt{\frac{1 + E_2 I_2/E_1 I_1}{1 + \mu_2/\mu_1}} = w\sqrt{\frac{1 + E_2 I_2/E_1 I_1}{1 + \mu_2/\mu_1}} \tag{5}$$

Finally, the ratio of the frequencies between the coated and bare TF is given in Eq.6 with its binomial approximation.

$$\frac{f'}{f} = \sqrt{\frac{1 + E_2 I_2/E_1 I_1}{1 + \mu_2/\mu_1}} \cong 1 + \frac{1}{2}\frac{E_2 I_2}{E_1 I_1} - \frac{1}{2}\frac{\mu_2}{\mu_1} \tag{6}$$

As a final point, Eq.6 can be explained in detail by including film thickness (*h*) and expansion of the second moment (*I*) for two kinds of cross-sections: circular and rectangular.

For a circular-cross-section- fork, the ratio of frequency shift to bare frequency is obtained in terms of *E* (Young's modulus), ρ (density), *r* (radius), and *h* (film thickness) as follows.

$$\frac{\Delta f}{f_{bare}} \cong \left(2\frac{E_{film}}{E_{bare}} - \frac{\rho_{film}}{\rho_{bare}}\right)\frac{h}{r} \tag{7}$$

Neglecting the effect of the flexibility of the film and electrode and when a small mass (considerably smaller than the beam mass) is attached to a tine of the quartz tuning fork [18], [19], [21] following equation can also be written where the subscript *eff* denotes the value of the effective length.

$$E_{film} = 0, \rightarrow \quad \frac{\Delta f}{f_{bare}} \cong -\frac{\rho_{film}}{\rho_{bare}}\frac{h}{r} = -\frac{1}{2}\frac{m_{film,\,eff}}{m_{bare,\,eff}} \tag{8}$$

Similarly, Eq.9 and 10 explain the same procedure for a rectangular-cross-section-TF where *a* and *b* represent the lengths of the rectangular cross-section which are perpendicular and parallel to the plane of oscillation respectively.

$$\frac{\Delta f}{f_{bare}} \cong \left[(3a+b)\frac{E_{film}}{E_{bare}} - (a+b)\frac{\rho_{film}}{\rho_{bare}}\right]\frac{h}{ab} \tag{9}$$

$$E_{film} = 0, \rightarrow \quad \frac{\Delta f}{f_{bare}} \cong -(a+b)\frac{\rho_{film}}{\rho_{bare}}\frac{h}{ab} = -\frac{1}{2}\frac{m_{film,\,eff}}{m_{bare,\,eff}} \tag{10}$$

Mathematical calculations of QTF resonance frequency are generally made by spring modeling in the literature, and while the frequency shift of the film is calculated, Young's modulus of the film is generally neglected and only its mass is taken as the basis. If the coated film is elastic, the contribution of flexibility has not been mentioned much [19], [22]. In this study, it was investigated how a flexible film coating on the QTF surface would affect the frequency shift. The equations we proposed above to calculate the frequency of the QTF are those we developed and presented with a different perspective (Euler–Bernoulli beam theory). Under the assumption of homogeneity, these equations can also be interpreted depending on the thickness of the film. Different from the equations in the literature, in particular equations 7 and 9, it will be possible to calculate the elasticity modulus of the film directly. However, as seen in equations 8 and 10, when the elastic modulus is neglected, only the mass-dependent equation valid in the literature is assumed.

3 RESULTS AND DISCUSSION

In this study, QTF surfaces were coated layer by layer for 2 min. in a plasma device. Each QTF was coated 10 times and frequency differences that were measured for each coating step. For each QTF and each 2-min. plasma deposition duration, Δf measurement results are given in Table 1.

The effective mass of the bare QTF (of one prong) [20] can be calculated from $m_{bare,\ eff} = \rho abL_{eff}$ where a, b, L_{eff} are thickness, width, and effective length, respectively (a: 0,585 mm, b: 0,248 mm, L_{eff}:3,9364 mm). ρ is density (2,65×10^3 kgm^{-3}) of the quartz. Assuming that Young's modulus contribution on frequency shift and the film thickness can be ignored [17],[19], [22] when Eq.10 (in literature) is applied to the frequency shift data, the mass of the film coated on the effective length ($m_{film,\ eff}$) of one of the prongs are calculated. The coated mass and time graph on QTF1 is shown in Figure 1. Results indicate that 22.14 nanograms of chemical are deposited on the effective length per minute in our experiment. This corresponds to 3.42 nm average film thickness per minute. Similarly, the same procedures are applied on QTF2 and Figure 2 is obtained. A mass of 20.76 nanograms is deposited per minute and 3.17 nm average thickness per minute is seen. In addition, approximately 92 ng of mass accumulation per 1 Hz was obtained.

If we look at the frequency shift results obtained with the plasma coating, a result close to linear was obtained depending on time. R-square (R_1-square =0.9743 R_2−square= 0.9249) tells us, that when Eq.10 (equation in the literature) is applied to the frequency shift data, Eq.10 and experimental data are more than 90 percent in agreement. This indicates that there are strong linear relationships between the most widely used mathematical model in the literature and the experimental data for both QTFs coated with layer-by-layer plasma. As can be seen, only the mass-dependent equation used in the literature gives satisfactory results when QTF is coated with a film with a low Young's modulus (11-MUDA). We consider that this equation is satisfactory for homogeneous coatings made with films with low Young's modulus, but not good for coatings made with films with high elastic modulus. In this case, we think that the equations 7 and 9 that we have developed and proposed will be valid.

4 CONCLUSION AND FUTURE PERSPECTIVE

A device that enables the measurement of frequency shift due to mass change with QTF has been developed by our research group and a patent application has been made (for single QTF measurement. Patent no: TR 2016 05031 B). In this study, we evaluated

Table 1: Δf values that are measured through the QTFs coated layer by layer.

Plasma deposition duration (min.)	QTF1Δf (Hz)	QTF2Δf (Hz)
2	0,5	0,5
4	1,5	1
6	1,5	2
8	2	2,5
10	2,5	2
12	3	2,5
14	3,5	3,5
16	3,5	3
18	4,5	4
20	4,5	4,5

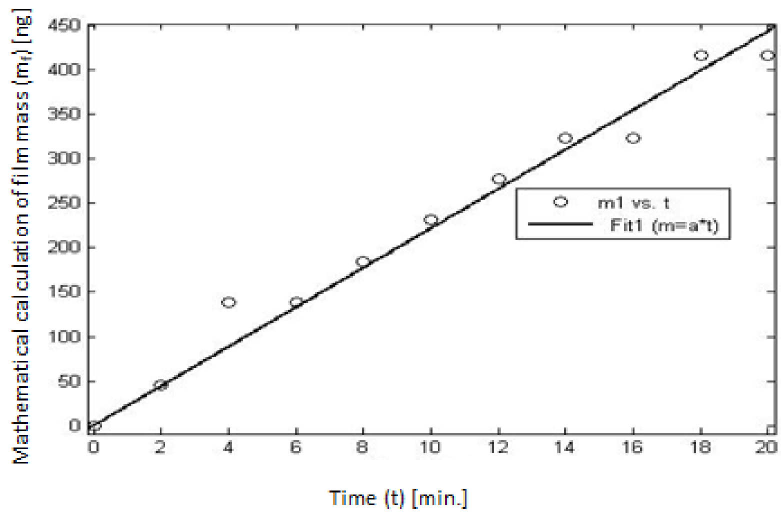

Figure 1: Mass of the film deposited on the effective length of QTF1 in time. Linear fit gives the output mf=22.14 ng -per minute with an R_1-square value of 0.9743.

the device by performing preliminary experiments in a plasma environment.

The QTF surface was coated with MUDA-11, a material with a low Young's modulus, and the theoretical calculation of the film thickness was examined for consistency with the equation, which is widely used in the literature and depends only on mass. When the equation, which is based only on mass and is widely used in the literature, is applied to the QTF frequency shift data, it has been observed that there is a high agreement between the experimental data and this equation. For the future, it aims to determine the film thickness values of our experimental data with AFM (Atomic Force Microscopy) or 3D Laser Scanning Microscopy and to compare the calculated thickness values with the measured thickness values and examine their consistency. In addition, it is aimed to cover the QTF with films with high Young's modulus and to investigate the contribution of Young's modulus of the film to the frequency shift and film thickness with the equations we proposed. For cases where Young's modulus cannot be neglected, the frequency shift of the film's Young's modulus and its contribution to the film thickness will be directly found, so we think that more accurate results can be obtained. Experimental thickness values obtained by AFM or 3-D Scanning Laser Microscopy of QTFs coated with high Young's modulus film will be compared with the theoretical thickness values obtained from the proposed formulas, and the consistency of experimental data with theoretical data will be examined. It should be noted that since the equations we propose are valid for the case of homogeneous coating of the QTF, it is necessary to control the homogeneity of the coating on the QTF surfaces along a single line. As a result, we think that the device we developed has the potential to be used in film thickness studies in the future and can be used as a more sensitive technique instead of QCM used for this purpose before. In addition, with the QTF multi-measurement system we are developing, it will be possible to measure not only from one region but from more than one (2,3,4) region within the coating. In this way, the average values with the values obtained from many QTFs will be determined and more accurate results will be obtained.

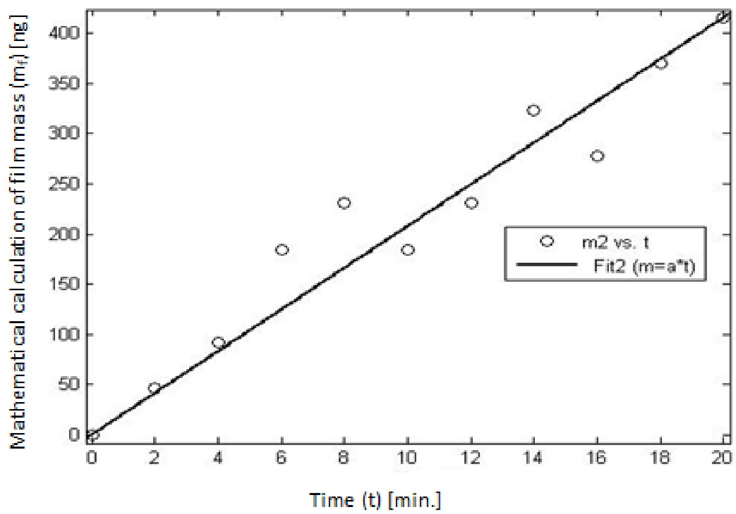

Figure 2: Mass of the film deposited on the effective length of QTF2 in time. Linear fit gives the output mf=20.76 ng -per minute with an R_2 -square value of 0.9249.

For this reason, we think that the thickness measurements of thin films coated on biomaterial surfaces can be made more precisely with the methodology we propose.

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The patent application for this work is pending (for multiple QTF measurements). Patent application no: 2022/018799

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