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Sensing characteristics of high-frequency shear mode resonators in glycerol solutions☆

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Abstract

Film bulk acoustic resonators (FBAR) vibrating in shear mode and operating at around 830 MHz have been fabricated. They were tested in air and water–glycerol solutions. With a sensitivity of 1 kHz cm^2/ng, these devices are attractive for gravimetric sensing applications. The FBARs are solidly mounted on acoustic mirrors and use 16° *c*-axis inclined ZnO thin films realized by reactive sputtering. An analysis of the performance in liquids with different viscosities has been done and the effect on quality factor and resonance frequency has been shown. Effective coupling coefficients K^2_{eff} of up to 1.7% and quality factors of up to 380 and 199 were determined in air and deionized water, respectively. The obtained characteristics are sufficient for gravimetric sensing applications in liquid environments and the FBARs can be used as high frequency viscosity sensors for liquids of viscosities up to 10 mPa s.
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1. Introduction

There have been many efforts to make use of bulk and surface generated acoustic waves for the development of sensors [1]. Quartz crystal micro-balances (QCM) and surface acoustic wave (SAW) devices have been utilized as bio-chemical gravimetric sensing devices in liquid environments [2,3] and for viscosity sensing [4,5]. Both concepts show limitations regarding the ability of downsizing as well as the integration with CMOS circuitry. Recently, film bulk acoustic resonators (FBAR) have also been proven to be applicable for gravimetric bio-chemical measurements in air and in liquids [6,7]. A typical FBAR consists of a thin piezoelectric film such as ZnO or AlN sandwiched between two metal layers. Since the sensor uses standing waves in the direction perpendicular to the substrate surface, the lateral sensor dimensions can easily be downscaled to 10 μm × 10 μm. FBARs constitute an attractive device for cheap, disposable and highly integrated sensor arrays. Furthermore, they are expected to show high mass sensitivities in the order of several kHz cm^2/ng as their operating frequencies can easily reach the GHz range.

In this article we present solidly mounted shear mode FBARs made of *c*-axis inclined ZnO. The FBARs are realized on acoustic mirrors functioning similarly to Bragg reflectors. Unlike the longitudinal wave mode where deflections perpendicular to the resonator surface radiate energy into the liquid, the shear bulk wave mode allows an operation with less damping effects. This has been demonstrated numerous times with AT-cut quartz crystals. However, compared to QCM devices, where typical resonance frequencies are in the range of 5–20 MHz, FBARs operate at much higher frequencies. The damping effects in liquids have a negative impact on the device *Q*-factor, which in turn increases the FBAR's noise equivalent mass change. Furthermore, the resonance frequency of the device is expected to decrease with increasing viscosity [8]. This fact can be exploited to realize viscosity sensors based on FBARs and has already been shown with preliminary measurements in deionized water [9]. In this paper a closer analysis of the performance in liquids

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of different glycerol concentrations with varying viscosities will be done and the effect on Q-factor and resonance frequency will be evaluated.

2. Mass sensing characteristics

FBARs can be used as bio-chemical or gas sensors based on the gravimetric principle [6]. In that case, the most important characteristics are selectivity, sensitivity and noise equivalent mass change [10]. The selectivity of the sensor to certain molecules is determined by the bio-chemical coating on the resonator surface.

The mass sensitivity is determined from the resonance frequency shift of the FBAR with mass changes at the resonator surface. For a simple QCM device the relation between the surface mass density μ (mass per area) and the resonance frequency f_0 can be described by the Sauerbrey equation holding for thin and rigid layers [11]. The absolute mass sensitivity s (the frequency shift per unit mass density change) is in that case proportional to the square of the resonance frequency:

$$s = \frac{\Delta f_0}{\Delta \mu} = -\frac{2}{v_{\mathrm{ac}}\rho} f_0^2 \tag{1}$$

where v_{ac} and ρ are the acoustic velocity and the density of the quartz plate, respectively. This relationship holds approximately true for FBARs. However, when taking into account the multiple layers of the FBAR, a more precise analysis of the sensitivity must involve simulations. In fact, differences of 80% to the Sauerbrey formulation could result if the exciting electrodes have a similar thickness than the piezoelectric layer [12]. For the FBARs presented in this paper, with operating frequencies of around 830 MHz, simulations using a one-dimensional transmission line model have shown that the sensitivity is around 1 kHz cm^2/ng, which is much higher than typical sensitivities of QCMs. This mass sensitivity does not change considerably in liquids. However, it could be influenced by the frequency shift due to varying viscosities, which in consequence must be known precisely.

In contrast, the noise equivalent mass change or detection limit μ_{r}, which is a measure of the smallest detectable surface mass density, depends on the device quality factor Q, which is adversely affected during operation in liquids. Assuming that the FBAR oscillator has a phase noise $\Delta\varphi$, the noise equivalent mass change μ_{r} is given by

$$\mu_{\mathrm{r}} = \frac{f_0}{2} \frac{\Delta\varphi}{sQ} \tag{2}$$

which results from formula (1) and the definition of the Q-factor from the slope of the FBAR impedance phase $\angle Z$ at the series or parallel resonance frequencies f_{s} or f_{p} [13]:

$$Q_{\mathrm{slope}}|_{f_{\mathrm{s,p}}} = \frac{f_{\mathrm{s,p}}}{2} \frac{\partial \angle Z}{\partial f_{\mathrm{s,p}}} \tag{3}$$

For QCM, typical values for μ_{r} are around several ng/cm^2. For FBARs operated in air, Q-factors lie between 300 and 1000 [14], which is much lower than typical values for quartz resonators. Nevertheless, due to the much higher oscillation frequencies of FBARs, detection limits comparable to QCM systems can be achieved. This can be easily recognized by considering formula (2), which shows that the noise equivalent mass change is inversely proportional to the sensitivity times the Q-factor. As liquids can sustain acoustic longitudinal waves, FBARs vibrating in the longitudinal mode dissipate energy into the liquid. The Q-factor drops to about 10, causing the noise equivalent mass change to jump to unacceptable high levels. On the contrary, the propagation of acoustic shear waves is barely supported in liquids. Thus, higher Q-factors and a lower noise equivalent mass change should be achieved. Nonetheless, with high viscosity values, it is expected that the Q-factor will drop too much to keep using the FBARs as bio-chemical sensors. Both the resonance frequency shift and the Q-factor reduction will be analyzed in the following paragraphs.

3. FBAR realization and characterisation in air

FBARs with an area of 200 μm × 200 μm have been fabricated. A schematic cross section of the FBARs is shown in Fig. 1(a). The active area of the device is determined by the area of the 100 nm thick top signal Pt electrode. The FBARs have fundamental shear mode resonance frequencies around 830 MHz. The high and low acoustic impedance Pt–ZnO layer pairs between the Si wafer and the bottom electrode constitute an acoustic Bragg reflector preventing the acoustic wave from penetrating into the substrate. The thicknesses of the different layers were calculated using a one-dimensional transmission line model based on the acoustic shear impedances. The mirror layers must have thicknesses of a quarter-wavelength; consequently, thicknesses of 500 and 840 nm were calculated for the Pt and ZnO layers, respectively. The 500 nm thick Pt bottom electrode is also the uppermost mirror layer. The active piezoelectric ZnO layer has a thickness of 480 nm.

Using ZnO thin films, shear modes can be excited when the c-axis is inclined with respect to the surface normal [15]. A c-axis inclined ZnO film was deposited reactively from a Zn target with a modified dc pulsed magnetron sputtering equipment. The sputtering method was optimized to produce highly insulating ZnO thin films with inclinations ranging from 0° to 16°. Details about this sputtering process using an additional blind between the substrate and the target have been published elsewhere [16]. For the ZnO films in the FBARs examined here, a c-axis inclination angle of up to 16° was determined by X-ray diffraction. At this inclination, longitudinal mode and shear mode can be excited with comparable electromechanical coupling constants. Fig. 1(b) shows a scanning electron microscopy micrograph of the cross-section of a fabricated FBAR. Inclined columns can be recognized in the uppermost ZnO layer.

The electro-acoustic characterization of the solidly mounted FBARs was carried out by one port measurements of the reflection coefficient S_{11} using a HP 8513A Network Analyzer. By fitting the measured data to a Butterworth–Van Dyke (BVD) model (Fig. 2), the series and parallel resonance frequencies f_{s} and f_{p}, effective coupling coefficient K_{eff}^2 and device Q-factor

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