Substrate texturing for homogeneous deposition of tilted c-axis AlN films for shear mode operation

M. Sierra-Zapata, M. Clement, T. Mirea, J. Olivares, and E. Iborra

Abstract—We propose two methods to improve the growth process of AlN films displaying the c-axis uniformly tilted, intended for the excitation of shear modes in acoustic resonators. The two methods aim at modifying the surface topography of the substrates in order to produce a family of parallel planes offering a suitable orientation for the subsequent growth of uniformly tilted AlN microcrystals. The first method is based on an ion milling process over rough substrates. Rough substrates, obtained by depositing a W layer on sputtered porous silicon oxide layers, are bombarded with a wide beam of Ar ions impinging on the sample surface at an angle of 65°. All treated surfaces lead to increased mean tilt angles of the subsequently grown AlN films, which in turn improves the coupling factor of the shear mode resonators. The second method consists in transferring the topography of a blazed diffraction grating to a photoresist layer spun over metal substrates. The photoresist pattern is then transferred to the substrates by a non-selective directional reactive ion etching process with SF₆.

Keywords— *BAW resonators; shear mode resonator; tilted grains AlN*

I. INTRODUCTION

Gravimetric sensors based on electroacoustic resonators with surfaces functionalized to promote the binding of targeted species are good candidates for biological sensing in liquid environments. Compared with quartz crystal microbalances (QCMs), resonators based on AlN thin films offer the possibility of achieving greater sensitivities, since they can be operated at frequencies in the range of GHz by reducing the thickness of the piezoelectric film [1].

For in-liquid applications, it is advisable that gravimetric sensors based in bulk acoustic wave (BAW) resonators operate in the shear mode, which radiates less acoustic energy to the liquid medium than the longitudinal mode. To excite shear modes in piezoelectric AlN films a component of the electric field must be normal to the c-axis, to produce shear displacements by virtue of the d_{15} piezoelectric coefficient. Two electrical configurations have been envisaged to achieve such excitation. The first one, called lateral excitation, is achieved by applying an electric field between two coplanar electrodes defined on top of a highly c-axis oriented film. This geometric arrangement would provide, according to previously reported works [2,3], a straightforward excitation of the shear

mode. The second configuration is based on the conventional excitation of AlN films using two electrodes placed on each side of the piezoelectric layer, but in this case, the AlN is made of microcrystals uniformly tilted with respect to the normal of the film, and this to the electric field [4]. This solution has proven to be far more effective than the lateral excitation [5]. It has been reported that tilt angles ranging from 20° to 45° provide the greatest values of the electromechanical coupling factor for the shear mode (k_s^2) [6]. Among the different techniques developed to achieve AlN films with tilted grains [7-9], off-axis sputter deposition over rough under-layers turns out to be the most effective [8]. In these kind of substrates, when the flux of atoms impinging on the different inclines is uniform, AlN grains tend to grow normally to the slopes in equal number in all directions, giving rise to a randomly-oriented AlN layer with averaged null piezoelectric response. However, if an impinging direction is encouraged (e.g. by displacing the substrate with respect to the target axis), the crystals will grow faster on the inclines most exposed to the atom flux and their mean tilt angle, normal to those planes, will be more uniform [9].

Two possible approaches can be used for controlling the uniformity and characteristics of these inclines. The first consists in carefully controlling the roughness of layers of the device that are closer to the active AlN film, either the bottom electrode or the uppermost layer of the acoustic reflector, in the case of solidly mounted resonators (SMRs). The second method involves the addition of a polycrystalline seed layer showing a controlled preferred orientation and exhibiting facets with the desired tilt angle [4, 10]. Both methods suffer from a significant lack of homogeneity in the basal orientation of the inclines, leading to some dispersion in the direction of the AlN microcrystals.

The aim of this paper is investigate new procedures to homogenizing the orientation of the substrate inclines for the subsequent growth of AlN films with homogeneously tilted grains. We propose two approaches to achieve this goal. The first one, hereafter “sculpturing”, consist in pre-treating the substrates by Ar-ion milling at incident angles close to 65° in order to sculpture the roughness of their surface before AlN deposition. The second approach, hereafter “nanoimprinting”, involves nanoimprinting a photoresist layer spun over the bottom electrode of the SMR and etching it subsequently

through a directional and poorly selective reactive ion etch (RIE) process, in order to transfer a saw-tooth-like profile to the substrate surface.

II. METHODS

The test substrates were $2 \times 2 \text{ cm}^2$ silicon dies covered with a $\lambda/4$ acoustic reflectors made of alternating SiO_2 and Mo films as low and high acoustic impedance layers, respectively. The targeted resonant frequency was around 2.5 GHz for the longitudinal mode and 1.4 GHz for shear mode. The uppermost SiO_2 layer of the reflector consisted in a sputtered porous layer displaying a mean roughness of around 4 nm. As bottom electrode we used sputtered tungsten (W) films or evaporated iridium (Ir) films.

Before AlN deposition, some samples were subjected to a roughness sculpturing process. These samples were bombarded by ion milling with an extended 100 mm in-diameter Ar-ion beam produced by a Gencoa IM75 ion gun of variable ion energy and current. Other samples were subjected to the nanoimprinting process. First, they were covered with a photoresist layer, which was subsequently nanoimprinted on the center with a PDMS replica of a $12 \times 12 \text{ mm}^2$, 1200 lines/mm diffraction grating having a blaze angle of 26° . The samples were then etched by CHF_3/SF_6 -based reactive ion etching (RIE) process adjusted to reduce the etch selectivity between the photoresist and the underlying substrate. This method allowed us to transfer the imprinted profile to the W bottom electrode or the SiO_2 uppermost layer of the SMR.

The AlN layers films were then sputtered in ultra-high vacuum system from a 150 mm in-diameter Al target in Ar/N_2 (40:60) mixtures. The substrates were displaced 5 cm from the center of the substrate holder, placed 5.8 cm apart from the target. The tilt angle of AlN grains was routinely derived from the position of the quasi-LO IR mode in the infrared reflectance spectra measured by Fourier transform IR spectroscopy (FTIR), using the method described in [11, 12]. Some samples were also assessed by measuring the XRD pole figure of the (00·2) reflection. Both measuring methods yielded similar values of the c-axis tilt angle.

BAW resonators were produced by adding a Mo top electrode to the whole structure. They were characterized by deriving their electrical impedance from the measurement of the reflection coefficient (S_{11}) performed with a network analyzer between 100 MHz and 5 GHz.

III. RESULTS AND DISCUSSION

A. Texturing the roughness of the substrate

Opposite to the growth of highly c-axis oriented AlN films, which requires polishing the surface of the acoustic mirrors, tilted AlN films can be achieved by taking advantage of the native roughness of the substrates combined with an off-axis sputter deposition. However, if this surface is not previously conditioned, the resulting AlN film displays a significant dispersion in the tilt angle. Trying to improve the uniformity of the inherently rough surface of an acoustic mirror, resulting from the successive growth of the different composing layers,

we decided sculpture the surface of the W bottom electrodes of several devices by ion milling them at 65° with respect to the surface normal at different ion energies and currents. During the process a part of each sample was intentionally masked with a glass plate so that the subsequent AlN film grew simultaneously on treated and not-treated areas. After the ion milling the samples were immediately loaded in the sputtering chamber for the off-axis AlN sputter process. The position of the Al-LO peak in the IR reflectance spectra measured in the sculptured part of the samples was systematically shifted toward greater wavenumbers, revealing that the sculpturing process tended to increase the tilt angle of the samples, to a greater or lesser extent.

Figure 1 shows the tilt angle derived from FTIR measurements of AlN films grown on W electrodes sculpted for 20 minutes under different experimental conditions. It is important to note that the data of figure 1 correspond to sample dies coming from the same 100 mm silicon wafer covered with a five-layer SiO_2/Mo -based acoustic reflector. This is so because samples coming from different substrates give rise to strong variations in the tilt angle, due to the uncontrolled roughness profiles of the acoustic reflectors from run to run. Results of figure 1 reveal that there is a threshold voltage (1050 V) above which the ion-milling process has a significant effect in the surface conditioning that promotes the growth of more tilted AlN films. Additionally, for voltages values above the threshold, lower ion currents induce greater tilt angles, which suggests that a too high bombardment may produce a smoothing of the rough surface.

Fig. 1. Tilt angle of AlN films grown over ion milled W substrates as a function of the ion current density and the Ar ion accelerating voltage

However, despite the evident increase of the tilt angle, the electric characterization of the resonators did not show a clear improvement in the performance of the shear modes, being impossible to find a trend between tilt angle and electro-mechanical coupling factor k_s^2 . All the results obtained in a large number of samples treated under different conditions suggested that the uncontrolled roughness profiles of the reflectors are hardly homogenized by an ion-milling process. Moreover, the lower values of k_s^2 , obtained in some samples after the ion milling treatment pointed out that, in some cases, the latter might even induce damage in the sample, leading to AlN films of lower piezoelectric activity.

B. Transfer of slopes by nanoimprint and RIE

To achieve greater tilt angles and improve uniformity and reproducibility of the engineered surfaces, we developed a two-step profile transfer method based on, first, nanoimprinting a saw-tooth profile on a thin photoresist layer (400 nm thick) using a stamp made of PDMS, and then transferring the profile to the substrate. The actual master for the stamp was a 1200 lines/mm ruled diffraction grating with a blaze angle of 26° . The transfer method was a RIE process based on an SF_6 plasma for W layers and on CHF_3 and SF_6 mixtures for SiO_2 layers. The etching processes were tuned to obtain the same etch rate for the resist and the layer underneath. Figure 2 shows a scheme of the etching process at different moments.

Fig. 2. Scheme of the transfer etching process. (1) starting profile; (2) moment just before starting substrate etching; (3) same etch rate for resist and substrate maintain the geometry; (4) end of the process.

Similar profiles of the surface topography were obtained when transferring the pattern directly to the W bottom electrode or when transferring the pattern to the SiO_2 outermost layer of the acoustic reflector before the growth of the W electrode. Fig. 3 shows two AFM 3D images of the topography of the W top electrode. Picture a) corresponds to a W substrate directly subjected to the profile transfer process and picture b) to a W layer grown on top of a nanoimprinted SiO_2 layer.

Fig. 3. AFM image of the surface of a) W film patterned through the two-step nanoimprinting process and b) W film deposited on a textured SiO_2 layer.

AlN films grown on top of W films textured through the nano-imprinting process tended to grow normal to the substrates, regardless of the surface topography. This is due to the fact that AlN microcrystals grow with the (00-2) planes

parallel to the (110) planes of the W grains, which always grow parallel to the surface when deposited by sputtering, regardless of the sputtering conditions. In fact, we varied the W sputter parameters and sample position in the substrate holder to investigate the effect of the directionality of the sputter process but we achieved always the same results. To solve this problem, we had to introduce a decoupling layer to prevent AlN grains from growing with the c-axis normal to the (110) planes of W. We tried with SiO_2 (without success) and with AlN thin layers sputtered under low energy conditions -low power (600 W) and high pressure (5 mTorr) - to promote the growth of randomly oriented AlN microcrystals. This way we succeed in “erasing” the memory of the surface and growing AlN films with the c-axis normal to the W layer inclines, as shown in the sketches of figure 4. The thickness of this AlN thin layer played an important role on the tilt angle of the subsequent AlN films as shown in figure 5.

Fig. 4. Sketch showing the mechanism that prevents the AlN grains from growing normally to the surface on engineered W films. a) normal growth on W inclines and b) growth on W slopes after erasing the surface memory via a poly-AlN layer

Fig. 5. Tilt angle of the piezoelectric AlN films grown on top of engineered surfaces as a function of the thickness of the poly-AlN film.

With the help of the poly-AlN decoupling layer we were able to produce AlN films with uniformly inclined microcrystals with mean tilt angles close to 30° from the normal, as shown in the XRD pole measurements of figure 6. These plots reveal that there is clear improvement in the uniformity of the tilt angle after the conditioning of the surface topography, which is additionally shifted to higher values (around 30°).

Fig. 6. Pole figure of the (00·2) AlN XRD reflection in non-imprinted and imprinted areas of a same sample.

Finally, we show in figure 7 the values of the electromechanical coupling factors achieved in the resonators subjected to surface conditioning. The shear mode resonators achieved by the two methods have coupling factors greater than those displayed by non-conditioned SMRs, reaching values close to 3%, similar to those obtained only by adding a miss-oriented thicker AlN seed layer on smooth substrates [10].

Fig. 7. Variation of the coupling factor of longitudinal and shear modes as a function of the AlN grain tilt angle.

IV. CONCLUSIOTNS

We investigate two new methods to engineer the bottom electrodes of SMRs in order to achieve tooth saw-like profiles that promote the growth of tilted AlN films. The first method, based in texturing the inherent roughness of the acoustic mirror surface by ion milling it at high angles (65°), leads to an increase in the tilt angle of the grains of the subsequently

grown AlN films, but does not improve their piezoelectric activity. The second method, based on sculpturing the surface of the bottom electrodes in a saw-tooth pattern throw a two-step nanoimprinting-RIE process does not promote by itself the growth of tilted films. The influence of the metal substrate crystallinity prevails over that of the substrate topography. A randomly oriented AlN seed layer prevents the AlN films from growing along the preferred orientation of the metallic surfaces. Films grown of imprinted samples display improved uniformity of the tilt angle, which reaches values of 30°.

ACKNOWLEDGMENT

This work was partially supported by Ministerio de Economía y Competitividad del Gobierno de España through project MAT2013-45957-R.

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