Reactive sputtering of AlScN thin films with variable Sc content on 200 mm wafers

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Abstract— Sc-doped AlN polycrystalline films are attractive active layers for high frequency (GHz range) acoustic resonators owing to the significant enlargement of the AlN piezoelectric activity with the increasing Sc content. To sputter homogeneously doped AlScN films on 200 mm Si wafers we use a configurable cathode containing a variable number of embedded Sc pellets to fine tuning the Sc content in the films. The method was implemented in an Endeavor-AT™ cluster tool from OEM Group, adapted for sputtering on 200 mm wafers. 1 μ m thick AlScN films with uniform Sc content (around 7 at.%), high crystal quality and good piezoelectric response have been sputtered over 200 mm production-level wafers.

Keywords—AlScN films; 200 mm silicon substrates; variation of Sc content.

I. INTRODUCTION

5G technologies require wider bandwidths and higher communication speeds for the new era systems. Part of the bands will be allocated below 6 GHz where electroacoustic filters based on piezoelectric thin films can play an important role. Other bands of higher frequencies (27–40 GHz, and 64–71 GHz) will require the use of microstrip or cavity microwave filters, which size is being reduced for application in mobile devices [1]. Although most of the tools needed for implanting the 5G technology are already mature, significant amount of work is still required in the filter side in order to fulfill the demanding specifications. To tackle the lower frequency bands resonators based on Sc-doped AlN films are being increasingly considered owing to the significant increase of their electromechanical coupling, which can double that achievable with pure AlN mature technology. But not only does future communication electronics require significantly improving the performance of the piezoelectric active films; other applications in the microelectromechanical devices area demand increasing the piezoelectric activity of thin film piezoelectric materials [2].

Since Akiyama [3] first synthesized Sc-doped AlN thin films in 2009, much effort has been devoted to the in-depth understanding of the actual properties of the new active material. Presently, manufacturers are trying to develop reliable deposition processes to bring the new material to production and incorporate it to the actual technology. Alloying Al and Sc presents some difficulties, which precludes the use of

compound targets with high Sc contents [4]. However, reactive sputtering of alloyed Al-Sc targets with moderate Sc contents is still the best choice so far to achieve good quality AlScN films of controlled composition. The possibility of varying the Sc content to modulate the piezoelectric response of the films would be very desirable for small volume production of microelectromechanical devices. This would require using alloyed targets of different compositions, which is not economically viable. Co-sputtering from two independent Al and Sc targets is a possible alternative technique although homogeneity over large substrates areas is hardly achieved.

In this paper we present the study of the AlScN thin films with around 7 at.% of Sc atoms obtained from a special target arrangement formed by two concentric ac-powered aluminum targets containing a variable and easily removable number of embedded Sc pellets. This method allows obtaining films with homogeneous and controllable composition on 200 mm silicon production wafers. The film properties were assessed by Rutherford back-scattering spectrometry, X ray diffraction, infrared reflectance and electrical impedance spectrometry.

II. METHODS

A. Sputter deposition method

The deposition system used in this work was an Endeavor-FX sputter tool recently introduced by OEM Group LLC. The tool contains a load-lock module, a transfer module equipped with a vacuum high-speed robot, a pre-cleaning process module equipped with a planarization rf module (PRF) plasma source, which allows substrate pre-cleaning, and up to four sputter modules equipped with S-gun magnetrons.

A special characteristic of the S-Gun magnetron is its dual concentric target arrangement shown in figure 1 [5]. Two independently controlled ring-shaped sputtering targets (diameters 7" and 11") are arranged so that both targets sit on the same vertical axis being, and the larger outer target is positioned above and outside the smaller inner target. A dc powered S-gun is typically employed for metal and alloy film sputtering, while an ac powered magnetron provides an ideal solution for reactive sputtering of non-conductive films such as nitrides, oxides, and oxynitrides. In the ac version, a 40 kHz alternating current power supply with a floating output is connected directly to the 7" inner and the 11" outer targets (Fig. 1). This cathode arrangement allows stable and arcing-free operation along with the ability to control intrinsic stress in

reactively sputtered films independently from other film properties [5]. The S-gun's capability to deposit highly c-axis oriented AlN films without wafer heating and biasing has been already demonstrated even with very thin thickness [6].

Fig. 1. The dual-target S-gun magnetron: (a) ac-powering schematic diagram and (b) process module arrangement.

The partial substitution of some Al atoms in the wurtzite AlN network by other metal atoms is a conventionally used method for the improvement of some material properties. Alloyed targets allow obtaining such types of films although it is not easy to study composition variations. The dual cathode arrangement of S-gun allows depositing ternary compounds by co-sputtering two single-element targets. However, this method does not succeed in achieving radial homogeneity in the film compositions. In this paper we describe a procedure that enables achieving radial homogeneity along the substrate and offers the possibility of changing the composition of the films in an easy way. This method consists in embedding pellets of the Sc doping material in the two concentric Al targets, as shown in figure 2. The number of pellets allows setting the composition of the film in a certain range whereas their spatial distribution between the inner and outer targets allows controlling the composition radial homogeneity in of the deposited film. With the current arrangement, films with up to 14 at.% of Sc can be deposited. By substituting the Sc pellets by Al ones, the Sc content can be reduced to zero. Higher Sc concentration could be achieved by using denser pellet distributions.

B. Material characterization

The structure of deposited films was characterized by x-ray diffraction measurements to explore the preferred orientation of the films (theta-2theta measurements) and the degree of

alignment of the microcrystals along the 00·2 direction (rocking curve around the wurtzite c-axis).

Fig. 2. The S-gun magnetron equipped with pure Al targets containing inserted Sc pellets

Intrinsic stress and in-wafer thickness distribution were routinely derived for all wafers by profilometry measurements.

The atomic composition of the films was assessed by Rutherford Back-Scattering (RBS) spectrometry. A 2.3 MeV beam of 4He^+ particles with a $5 \mu\text{C}$ dose was used for the analysis, measuring backscattered particles with a scattering angle of 170° and using 20 keV resolution detector. Figure 3 shows the typical RBS spectra across a wafer radius, indicating the signals corresponding to the different elements. RBS spectra reveal that the composition of the AlScN film is roughly homogenous across the thickness of the films.

Fig. 3. RBS spectra of a wafer along its radius. Al and Sc arrows point out to the signals corresponding to the surface of the AlScN film. The signal corresponding to the Mo bottom electrode is superimposed to the Al signal. The signal corresponding to the first Mo layer of the reflector appears at lower backscattered energies

Additionally, infrared absorption measurements in the reflectance mode (IRR) were carried out to analyze the fine structure of the longitudinal optical (LO) region of the reststrahlen band [7]. Figure 4 shows a typical IR reflectance spectrum of a $1 \mu\text{m}$ -thick AlScN film, showing the interference

fringes from which the film thickness was derived, and the reststrahlen band region (around 700 cm^{-1}) showing the characteristic LO absorption band at its edge.

Fig. 4. FTIR spectrum showing the interference fingers due to the multiple reflections in the film. The inset show a detailed view of the reststrahlen band with the LO band in its edge.

Finally, for electroacoustic characterization, we manufactured test bulk acoustic wave (BAW) resonators over simple acoustic mirrors made of five alternating layers of Al and Mo, which provided sufficient acoustic isolation. The quality factor, the material electromechanical coupling factor, the longitudinal sound velocity and the dielectric constant were derived by fitting the electrical measurements with Mason's model [8]. Electrical measurements consisted in the measurement of the reflection scattering parameter S_{11} by using a network analyzer, from which the electrical impedance spectrum was derived. In figure 5 a typical electrical impedance spectrum is shown.

Fig. 5. Electrical impedance spectrum of a typical resonator.

III. RESULTS AND DISCUSSION

The targeted composition of our films was around 7 at.% Sc films. To achieve it, we used 10 out of the 20 possible Sc pellets in the inner target and 14 out of the 28 possible Sc pellets in the outer target. Before the deposition of the films, the substrates were etched in PRF module (Ar plasma at rf power) and then pre-heated to about 350°C in the sputter module. In addition, the wafers were intentionally heated during the first stage of the deposition process (from 50 nm to 600 nm) to improve the structure of the growing films.

The structure of all analyzed films is wurtzite highly c-axis oriented. The plane spacing of the AlScN films, obtained from the (00·2) diffraction angle, was around 5.00 Å $\pm$ 0.005 Å, which is a value 5% higher than pure AlN. This indicates that

the increase in the piezoelectric activity, which will be commented below, is due to the deformation of the wurtzite structure. The full width at half maximum (FWHM) of the $\theta/2\theta$ diffraction peak of around 0.15° reveals large size microcrystals and uniformly distributed Sc atoms into the material crystal network. The FWHM of the rocking curve around the (00·2) reflection varies from 1.6° to 2° in all analyzed samples, indicating a very good crystal quality. Some AlScN films, deposited on the wafers with roughest Al/Mo acoustic reflectors, are those showing the widest rocking curves. Probably the roughness of the substrate is responsible of these high rocking curve widths.

RBS compositional analysis shows an atomic Sc content ranging from 6 at.% to 7.5 at.%, wafer to wafer, which is close to the targeted composition (7 at.%). This content is higher in samples deposited at the highest substrate temperature indicating better Sc incorporation to the films. Homogeneity across the 200 mm diameter of the wafer was always below the 10% of the nominal value, being the Sc content always slightly higher at the edges of the wafer. To avoid this, a restructuring of the Sc pellets into the targets described above should be undertaken.

Infrared absorption in the reflection mode was chosen as a sensitive and nondestructive method to determine the Sc content in the film. Usually the presence of different neighbor atoms induces slight variations in the bond energy between two atoms, which modifies the wavenumber of the associated vibrational modes appearing in the absorbance spectra. Sometimes, these variations are used to determine the amount of doping in the material but the wavenumber shift may also account for other effects, such as stress-induced deformation. In the case of AlScN films we found that there are two different absorption bands with close absorption wavenumbers, which can be attributed to Al-N and Sc-N bonds.

Fig. 6. IR reflectance spectrum of a typical AlScN layer and its decomposition in gaussian bands associated to Al-N and Sc-N bonds. A small non-assigned band is also present.

The position of these absorption bands varies with the Sc content in the films. This situation is not usual and for this material we can take advantage from it. Figure 6 shows the LO region of the reflection spectrum (depicted as absorbance) is shown for a pure AlN film and for an AlScN films together with their best fits with a combination of Gaussian functions. Pure AlN spectrum can be fitted with a single one Gaussian

band at 890.89 cm^{-1} while AlScN requires two bands at 862.11 cm^{-1} and 869.4 cm^{-1} , respectively.

The first one is assigned to the Sc-N bonds and the second one to the Al-N bonds. As in many other materials, their particular position in photon energy depends on the neighbors and then, if Sc content varies, these band positions also vary. This effect can be observed in figure 7 that highlights the shift in the position of these bands as a function of the atomic percentage of Sc atoms in the narrow range obtained in this work.

Fig. 7. Al-N and Sc-N associated absorption band positions as a function of the Sc at% in the films. Variations in Sc content is due to differences in deposition conditions and inhomogeneity inside each wafer.

Piezoelectric response of the material was estimated through the measurement of the electromechanical coupling factor (k^2) of BAW resonators derived from the fitting of the electrical impedance spectrum to the Mason's model [8]. This method allows obtaining a value of k^2 characteristic of the material and not of the geometry of the particular resonators if their quality factors (at resonant and antiresonant frequencies) are high enough (> 20). Although the acoustic reflectors used in this work were not optimized, the quality factor of the devices fabricated fulfill this requirement ($Q > 50$ in all cases) allowing achieving reliable values of material k^2 . In all measured wafers, the values of k^2 were greater at the edge of the wafer than at the center. This is not clearly related with the radial variation of the Sc content, nor to the variation of any other accessible parameter of the sputtering system. We attribute this variation to a difference in the ionic bombardment of the surface of the wafer during the deposition process. The use of the independent control of stress and other film parameters [5] make this possibility very likely. Ionic bombardment has other effects in the material like the growth of misaligned grains that lower the piezoelectric response [7,9]. Additional experiments were carried out to obtain films with uniform values of the k^2 , as shown in figure 8. The sound velocity of the longitudinal mode for AlScN films resulted to be slightly lower than that of pure AlN. Values ranging between 9200 m/s and 10800 m/s were estimated from the fittings of the resonator response. Similarly, we also found values of the material dielectric constant higher than that of pure AlN. The estimated values were ranged between the pure AlN value of 82 pF/m to 90 pF/m . The estimated values of these parameters (sound velocity and dielectric constant) are very dependent on the thickness of the AlScN layer. Thickness determination by optical profilometry show a variation of

around 2 % into the wafer by using a refractive index of 2.088. The thickness obtained by this method can be affected by other parameters making the estimated values of the sound velocity and dielectric constant to be affected by a significant error.

Fig. 8. In wafer radial variation of k^2 of a representative sample.

CONCLUSIONS

AlScN films were deposited by an original PVD method consisting in an AC excited double cathode containing variable number of pure scandium pellets embedded in a pure Al matrix. The number and distribution of pellets in the cathodes allow controlling the film Sc content and the composition uniformity in 200 mm in diameter production wafers. As an example 7 at.% Sc films were deposited under different deposition conditions, yielding reasonably good uniform distribution of Sc in each wafer (within 10% of variation) and a slight variation from 6 at.% to 7.5 at.% of Sc from wafer to wafer. Properties of the films are excellent under a structural point of view and can be improved by making more uniform the ionic bombardment, in their piezoelectric response.

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