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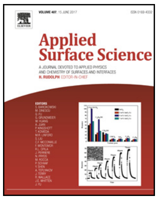

Microstructure and mechanical properties of stress-tailored piezoelectric AlN thin films for electro-acoustic devices

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ABSTRACT

Nanoindentation measurements along with atomic force microscopy, X-ray diffraction, and residual stress analyses on the basis of Raman measurements have been performed to characterize stress-tailored AlN thin films grown using reactive RF magnetron sputtering. The intrinsic stress gradient caused by the growing in-plane grain size along film thickness was minimized by increasing the N_2 concentration in the Ar/N_2 gas mixture during the growth process. The increase of N_2 concentration did not degrade the device-relevant material properties such as crystallographic orientation, surface morphology, piezoelectric response, or indentation modulus. Due to comparable crystallographic film properties for all investigated samples it was concluded that mainly the AlN crystallites contribute to the mechanical film properties such as indentation modulus and hardness, while the film stress or grain boundaries had only a minor influence. Therefore, by tailoring the stress gradient in the AlN films, device performance, fabrication yield, and the design flexibility of electro-acoustic devices can be greatly improved.

1. Introduction

Aluminum nitride (AlN) has been studied as thin film piezoelectric material for the fabrication of micro-electro-mechanical systems (MEMS) over the decades [1,2]. It has been proven to be advantageous material for electro-acoustic resonators in the area of radio frequency (RF) filters as well as for bio/chemical sensing applications [3,4]. Moreover, in combination with nanocrystalline diamond, it is a promising candidate for high quality factor acoustic filters for the next generation mobile communication devices [5,6].

Even though high quality AlN can be deposited using molecular beam epitaxy, or metalorganic chemical vapor deposition techniques [7,8], reactive magnetron sputtering is usually used for MEMS [9,10] which allows for low temperature processing. However, sputter deposited AlN films usually exhibit a pronounced intrinsic stress gradient along film thickness. Typically, the film stress is more compressive at the beginning of film growth and becomes less compressive or even tensile with increasing film thickness. Previous studies explain it as a result of lattice mismatch between the substrate and deposited film [11,12], coalescence of the individual crystal grains [13,14], decreasing grain boundary density along film thickness [15], or shadowing effects caused by the increasing surface roughness of the film [11]. The stress gradient has a negative influence on device performance, and can cause large curvature in free-standing microstructures [16]. Moreover, the quality factor and the acoustic phase velocity are also affected, as it was described for laterally vibrating micro-resonators [17]. In order to reduce the impact of the stress gradient advanced designs for suspended AlN beams have been proposed [18] and for membrane-based devices, such as Lamb wave resonators, designs with the membrane edges clamped to the substrate are possible [19]. However, this design limitation results in an increase of unwanted anchor losses due to radiation of energy into the surrounding substrate. In addition to device performance, there are also technological reasons for minimizing the stress gradient of

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Table 1
Sputtering parameters.

Parameter	Value
Target	Aluminum 5N, 200 mm diameter
Base pressure	$<10^{-5}$ Pa
Process pressure	8×10^{-3} mbar
RF-power	1000 W
Target-to-substrate-distance	59 mm

Table 2
Summary of the varied sputtering parameters for series (1)–(3).

Series	Set N_2 concentration (%)	AlN film thickness from ellipsometry (nm)
(1)	12	480
	22	480
	33	480
	50	480
(2)	22	240
	22	490
	22	750
	22	960
(3)	15–25	240
	15–45	470
	15–60	680
	15–67	870

sputtered AlN films: While the measured film stress, typically averaged over film thickness, is in a moderate range of less than 300 MPa [20], the stress can be very high at the substrate/film interface and at the film surface, leading to film cracking. Therefore, to improve the fabrication yield and to reach maximum design flexibility the stress gradient of sputtered AlN films should be carefully tailored. The total film stress in sputtered AlN thin films may be modified by adjusting the energy of the adatoms arriving on the film surface [21]. However, compensating the intrinsic stress gradient is more challenging, because the energy of the adatoms has to be changed during film growth gradually, for instance by increasing applied RF-bias [22], tuning the nitrogen concentration in the Ar/N_2 reactive atmosphere [13], or applying a heat treatment before and during the deposition [16,23].

In addition to compensating the intrinsic stress gradient, the structural and piezoelectric AlN film properties also need to be optimized to obtain maximal electromechanical coupling coefficient k_t^2. Moreover, the crystallographic film properties, such as degree of *c*-axis orientation and the tilt of the individual grains, greatly affect device performance, as it has been shown for AlN based film bulk acoustic resonators (FBARs) [24]. Finally, for analytical or finite element based modeling of devices, the total film stress and the Young's modulus of the AlN film are of particular importance [25–27].

Compared to the other works discussing the intrinsic stress gradient, the influence of the changing process conditions on AlN material properties has not been investigated in detail until now. The aim of this work was to analyze the influence of increasing N_2 concentration in the Ar/N_2 gas mixture during the growth of reactive sputtered AlN films on the intrinsic stress gradient, as well as on the structural, piezoelectric, and mechanical properties. By the comprehensive characterization of the AlN films it is shown that the changing process conditions did not degrade the above discussed device-relevant material properties.

2. Experimental details

AlN thin films were grown on boron doped silicon (001) wafers with a diameter of 100 mm and a resistivity of 10–20 mΩ cm in a magnetron sputtering system von Ardenne CS730S. The base pressure was $<10^{-5}$ Pa. Prior to deposition the Si wafers were wet-chemically cleaned in a mixture of $H_2SO_4 + H_2O_2 + H_2O$ followed by dipping in 1% HF for 30 s to remove the native oxide. After transferring the sample into the sputter chamber the Si substrate was *in-situ* sputter-etched for 120 s in low energy Ar plasma to improve the nucleation behavior of AlN on the Si substrate [28]. The AlN sputter process was performed in a reactive gas mixture of argon and nitrogen (Ar/N_2) and the total process pressure was kept constant independent of the total gas flow using a butterfly valve in front of the turbomolecular pump. The Al target was RF powered (1000 W, 13.56 MHz). The substrate heater temperature was set to 450 °C. The basic sputtering parameters which were kept constant for all samples are summarized in Table 1. Further details on the sputter equipment are described elsewhere [20].

In order to analyze the influence of residual film stress on device-relevant film properties, and in order to minimize the intrinsic stress gradient along film thickness, three series of samples have been grown (Table 2): (1) Samples with 12–50% N_2 in Ar/N_2 sputter gas mixture. The deposition time was adjusted to grow films with thickness of 480 ± 20 nm; (2) samples with film thickness of 240–960 nm, where all parameters except the deposition time were kept constant (N_2 = 22%); and (3) increasing N_2 concentration in the Ar/N_2 gas mixture during the growth for samples with film thickness in the range of 240–870 nm. The N_2 flow was maintained constant for all samples at 20 SCCM (SCCM denotes cubic centimeter per minute at STP) and the N_2 concentration (*i.e.* volume ratio of N_2 compared to total volume Ar + N_2) was tuned by adjusting the Ar flow. Here, mainly the N_2 ratio is influencing the film properties, whereas the change in total gas flow (at constant total pressure) has no significant influence on material properties, such as crystal quality or film stress [21,29]. Fig. 1 shows the variation of the N_2 concentration as a function of film thickness. Here, to equilibrate the film growth, the first 100 nm were grown with constant N_2 concentration of 15%. Subsequently the N_2 concentration was gradually increased to up to 67% for AlN film thickness of 750 nm, and then kept constant until the final film thickness was reached.

The thickness of the AlN films was determined by means of rotating-analyzer variable-angle spectroscopic ellipsometry (VASE™ of J. A. Woolam, Co.). A Cauchy-function with Urbach tail was used to describe the AlN dielectric function [30]. X-ray diffraction (XRD) $2\theta/\theta$ scan and 00.2 reflection rocking curve (ω-scan) measurements have been performed to examine the phase and crystallographic orientation. A commercial Berlincourt piezometer (PM300 from Piezotest) was used to measure the piezoelectric charge coefficient $d_{33,f}$. Film surface morphology, average grain size and surface roughness were investigated using atomic force

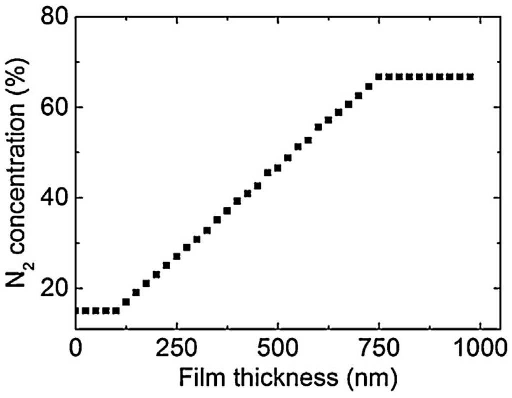

Fig. 1. Gradual increase of N_2 concentration in the Ar/N_2 gas mixture as a function of the AlN film thickness (series (3)).