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Mechanical and electrical properties of RF magnetron sputter deposited amorphous silicon-rich silicon nitride thin films

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ABSTRACT

Amorphous silicon nitride thin films in a thickness range of 40 to 500 nm are deposited onto (100) silicon wafers using radio frequency magnetron sputter deposition. Analysis of variance techniques are used to determine which deposition parameter has a significant impact on the film properties. The biaxial stress of the layers is found to be compressive independent of the plasma chamber pressure levels and to increase with increasing plasma power. The chemical composition of the films is silicon-rich, resulting in an index of refraction (IOR) of 2.55 independent of deposition conditions. Both IOR and X-ray photoelectron spectroscopy measurements indicate a nitrogen to silicon ratio in the range of 0.71–0.85. The etch rates for HF wet chemical etching and for CF_4:O_2 reactive ion etching are found to be much higher compared to direct current sputter deposited silicon nitride films with only a weak dependency on the deposition conditions. Temperature dependent leakage current measurements using Au/Cr/SiN_x/Si structures between 25 and 300 °C show two dominating leakage current mechanisms: ohmic conduction dominates at low applied electric field values below 0.1 MV/cm and Poole–Frenkel type conduction above 0.3 MV/cm. The extracted electrical parameters such as the activation energy or the barrier height are found to be nearly unaffected by the deposition parameters.

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1. Introduction

Silicon nitride (Si_3N_4 or more general SiN_x) thin films are used in a wide variety of applications in microelectromechanical systems (MEMS) due to their outstanding properties like high chemical resistance against many standard etch media [1], the ability to withstand high temperature manufacturing processes and excellent dielectric insulation [2].

A large variety of deposition methods has been developed due to the widespread integration of silicon nitride thin films in both front and back end of line device concepts, depending on various requirements such as thermal budget or hydrogen content. Chemical vapour deposition (CVD) techniques such as low pressure (LPCVD) [3,4], plasma enhanced (PECVD) [5–7] or catalytic chemical vapour deposition (Cat-CVD) [8] are among the most commonly used approaches. Drawbacks of CVD processes include high deposition temperatures in the range of 700 to 800 °C for LPCVD deposition [4], which makes the integration into a MEMS fabrication process challenging. PECVD processes allow the deposition to take place at more moderate temperatures ranging from room temperature up to typically 400 °C at the expense of a significantly increased hydrogen content in the deposited thin film [7]. Physical vapour deposition (PVD) techniques such as reactive sputter deposition using a polycrystalline silicon (Si) target or a ceramic Si_3N_4 target in a nitrogen-rich plasma proves to be a viable alternative approach. Commonly, radio-frequency (RF) sputter deposition is applied for film synthetisation, especially when using low conductivity targets like ceramic Si_3N_4 [9–13]. Besides this, direct current (DC) sputtering is also a feasible approach given a sufficiently high conductivity of the target, for example when using polycrystalline silicon [14,15].

Besides MEMS applications, silicon nitride thin films have been used together with silicon oxide since the 1960s as electrical insulation layers in microelectronics, resulting in extensive investigations into the electrical properties of SiN_x thin films over the last decades [9,13,16–21]. Most studies found the electric conduction mechanism in SiN_x thin films to be bulk-controlled [20,22,23], while in some cases indications for space charge limited conduction [24] or field emission [25] have been reported. Basically, those findings were dependent on the measurement procedure, the sample configuration, the film thickness and the applied deposition method as well as the deposition conditions.

In a previous study, DC sputter deposited silicon nitride thin films have been investigated with focus on their physical, chemical and electrical properties [14]. Given the mentioned influence of the deposition method and conditions on the electrical properties, it is reasonable to directly compare DC with RF sputter deposited silicon nitride in order to determine the specific advantages of each approach for MEMS device fabrication processes while using the same equipment for film synthetisation. In this work, the physical, chemical and electrical properties of RF sputter deposited silicon nitride thin films are investigated with respect to different deposition

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conditions like plasma power and chamber pressure levels as well as different film thickness values and directly compared to DC sputter deposited thin films.

2. Experimental details

The amorphous silicon nitride thin films studied in this work are deposited by RF magnetron sputtering onto double-side polished, single crystalline n-type (phosphorous doped) ⟨100⟩ CZ-silicon wafers. The resistivity of the substrate is >50 Ω·cm, the thickness is 350 ± 15 μm and the diameter 100 mm. Prior to deposition the substrates are cleaned in acetone and isopropyl alcohol, and the native oxide is subsequently removed using a short dip in buffered hydrofluoric acid (BHF), followed by rinsing in de-ionized (DI) water. Finally, the silicon surface was cleaned using an inverse sputter etching (ISE) step at a chamber pressure of 6 μbar and a power of 200 W in a pure argon atmosphere for 1 min.

The silicon nitride deposition as well as the ISE treatment is done in a *Von Ardenne LS730S* sputtering system using RF magnetron sputtering at 13.56 MHz. The ISE electrode has a diameter of 150 mm. The gaseous atmosphere during deposition is a mixture of argon (Ar, purity 6 N) and nitrogen (N_2, purity 6 N) with fixed flow rates of 20 sccm for argon and 60 sccm for nitrogen. The base pressure of the vacuum chamber before processing is $<10^{-7}$ mbar. A 150 mm diameter 6 mm thick polycrystalline silicon disc bonded to a copper plate and positioned 65 mm above the substrate serves as target. The RF plasma power P is varied between 300 and 900 W (this corresponds to a power density of approximately 1.5 – 4.5 W/cm^2 and a bias voltage U_B between 70 and 180 V respectively). The process chamber pressure p is varied between 3 and 9 μbar.

In order to investigate the influence of the deposition conditions (p, P) on properties like deposition rate R, index of refraction (IOR) n, biaxial film stress σ, material composition and etch rates r, samples with a film thickness $d = 500$ nm are deposited by adjusting the deposition times accordingly. The thickness values and refractive indices are determined using a *Filmetrix F20* spectral reflectometry system by measuring the broadband reflectance spectrum between wavelengths of 200 and 1100 nm. The biaxial film stress is measured with an *EH metrology MX203* capacitive wafer geometry gauge. X-ray photoelectron spectroscopy (XPS) is applied to determine the surface material composition using a *Thermo Scientific Theta probe* system. Fourier transform infrared spectroscopy (FTIR) measurements are taken using a *Bruker Tensor 27* infrared spectroscope providing a resolution of 4 cm^{-1} between wavenumbers of 400 and 4000 cm^{-1}.

Etch resistance is investigated in an *STS320* parallel plate reactive ion etching (RIE) reactor using a CF_4:O_2 gas mixture with parameters taken from [26], by wet etching in a 1:7 mixture of 49% aqueous HF and DI water and in a *Xactix e1* Xenon difluoride (XeF_2) plasmaless etching

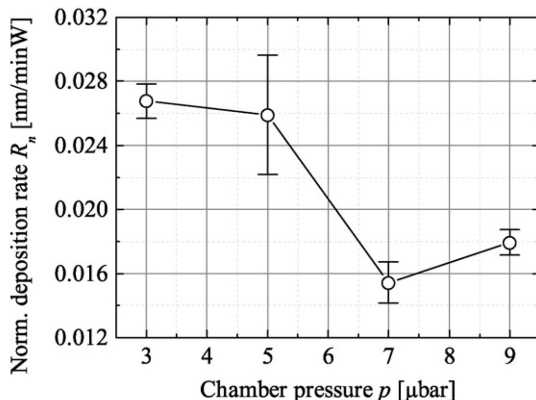

Fig. 1. Normalized deposition rate R_n for different chamber pressure levels.

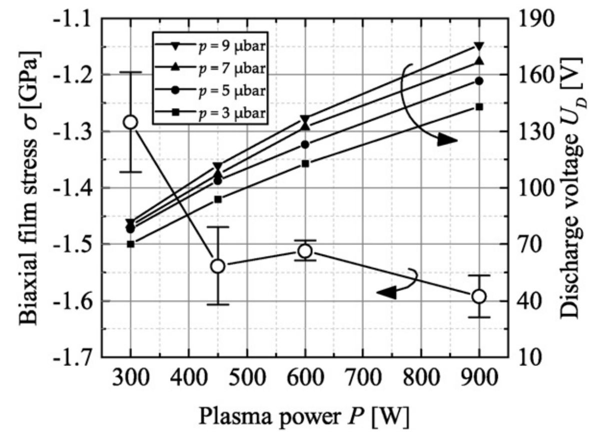

Fig. 2. Biaxial film stress σ and discharge voltage for different plasma power.

system (etch pressure 4 mTorr). Depending on the etch medium used, different etch times are used: 2 and 4 min for RIE, between 1 and 30 min for HF and 20 and 40 cycles of 30 s each for XeF_2 etching. The resulting etch depth is determined by measuring the change in film thickness before and after etching.

Samples for the leakage current measurements are fabricated in a similar way at four selected parameter sets (combinations of $p =$ 3 μbar and $p =$ 9 μbar as well as $P =$ 450 W and $P =$ 900 W) with varying film thickness ($d =$ 40, 100 and 300 nm). The silicon backside of all samples is coated with an 800 nm thick sputter deposited aluminium film for an enhanced electrical contact to the vacuum equipped wafer chuck serving as electrical ground. The electrical front side connection to the silicon nitride thin film is established by e-beam evaporation of 10 nm chromium as adhesion promoter and subsequently 200 nm gold. The front side metallization is patterned by lift-off to form an array of circular electrodes with a diameter of 1000 μm. The leakage current I at different applied voltages U and temperatures T is recorded on a temperature controlled wafer probe station (between 25 °C and 300 °C) *PM8* form *Süss Microtec* using an *Agilent B2911A precision source/measure unit*. The maximum electric field $E = U/d$ which is applied to the front side electrode pads using tungsten needles is $E =$ 0.5 MV/cm in both biasing directions. The measurement routine is described in more detail in [14].

3. Results and discussion

3.1. Physical and chemical properties

The deposition rate R increases linearly with the plasma power for $P \geq$ 300 W, as expected. At power values below 300 W, no significant sputter deposition takes place, which is attributed to an insufficient ionization rate of the gaseous atmosphere in the reaction chamber. The power normalized deposition rate is calculated as the slope $R_n = \partial R/\partial P$ and shown in Fig. 1. It decreases with increasing chamber pressure levels, which can be attributed to an increased collision rate of the ejected target atoms with the gas atmosphere in the reaction chamber. A similar behaviour has been observed for DC magnetron sputtered silicon nitride thin films [14]. The normalized deposition rate is lower compared to DC magnetron sputtering by a factor of about 1.5–2. This behaviour is called the deposition rate "paradox" and has been reported

Table 1
Chemical composition of samples deposited at $P =$ 600 W.

	Si	N	O	χ
$p =$ 3 μbar	51.0	43.1	5.9	0.845
$p =$ 5 μbar	51.3	43.5	5.2	0.848
$p =$ 7 μbar	51.2	43.8	5.0	0.855

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