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Mechanical and electrical properties of DC magnetron sputter deposited amorphous silicon nitride thin films

D. Dergez *, M. Schneider, A. Bittner, U. Schmid

Institute of Sensor and Actuator Systems, Vienna University of Technology, Floragasse 7, A-1040 Vienna, Austria

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ABSTRACT

Amorphous silicon nitride SiN_x thin films in a thickness range of 40 to 500 nm were deposited onto (100) silicon wafers using DC magnetron sputtering. The biaxial stress of the films was found to be tuneable in the range of −1300 MPa (compressive) to almost 0 by varying the plasma power density and the back pressure in the deposition chamber. The films were close to stoichiometric composition with x ranging from 1.27 to 1.34. The refractive index n decreases from 2.10 to 1.97 with increasing back pressure, indicating compositional changes in the thin films. This finding, however, was not confirmed by Fourier-Transform Infrared Spectroscopy measurements. On the other hand, lower wet chemical etch rates revealed a larger robustness of layers deposited at conditions where the mean kinetic energy of the sputtered particles is higher. Temperature dependent leakage current measurements using Au/Cr/SiN_x/Si MIS (metal–insulator–semiconductor) structures between 25 and 300 °C have shown that ohmic and Poole–Frenkel conduction mechanisms dominate the leakage current behaviour at electrical fields ranging up to 0.5 MV/cm. The extracted physical parameters such as the corresponding activation energies were found to be mildly affected by the deposition parameters.

1. Introduction

Silicon nitride (Si_3N_4) thin films are widely utilized in microelectronic and microelectromechanical applications, because they offer a large range of outstanding properties, such as excellent masking against oxidation [1], and certain etch media [2], the ability to withstand high-energy manufacturing processes, and their generally excellent insulation properties [3].

The widespread employment of silicon nitride thin films in both FEOL (front end of line) and BEOL (back end of line) applications led to the development of a large variety of available deposition methods depending on various requirements such as thermal budget or hydrogen content. Conventionally, silicon nitride thin films are deposited using chemical vapour deposition (CVD) methods, such as low-pressure (LPCVD) [4,5], catalytic (Cat-CVD) [6] or plasma enhanced chemical vapour deposition (PECVD) [7–9]. LPCVD requires high deposition temperatures typically ranging from 700 to 800 °C [4], thus hindering the integration of such films into some device fabrication processes. In contrast, PECVD processes offer reduced deposition temperatures depending on the type of plasma coupling, ranging from room temperature up to 400 °C [9] at the expense of increased hydrogen content. On the other hand, sputter-deposition using a ceramic Si_3N_4 target or a polycrystalline Si target in combination with a nitrogen-rich plasma is also a viable alternative. Typically, a radio-frequency (RF) discharge is applied for synthesis [10–14], but given a sufficient electrical conductivity of the target, direct-current (DC) magnetron sputtering is also a feasible approach [15,16].

Given the fact that SiN_x has been used as an electrical insulator since the 1960s in microelectronics, extensive investigations were focused on the electrical properties of SiN_x thin films for the past decades [10,13, 17–22]. Most studies concluded that the conduction mechanisms in thin SiN_x films are bulk-controlled [17,23,24], while some have found indications for space-charge-limited conduction (SCLC) [25], or field emission [26]. Commonly, these findings were not only dependent on the measurements structure, but also on the thickness of the films and the applied deposition method.

It is the objective of this study to investigate the temperature-dependent leakage current behaviour of DC magnetron sputter-deposited silicon nitride thin films of various thicknesses from room temperature up to 300 °C, in order to identify the dominating conduction mechanisms and to extract the corresponding physical parameters. Furthermore, the influence of the sputter deposition parameters (DC power and back pressure) on the resulting optical, mechanical and chemical properties of the SiN_x thin films is investigated, to link these results with those gained from the leakage current measurements.

2. Experimental details

The substrates used for the silicon nitride thin film deposition were double-side polished, single-crystalline silicon n-type (phosphorous doped) wafers, with a <100> orientation, a bulk resistivity of

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* Corresponding author.
E-mail address: david.dergez@tuwien.ac.at (D. Dergez).

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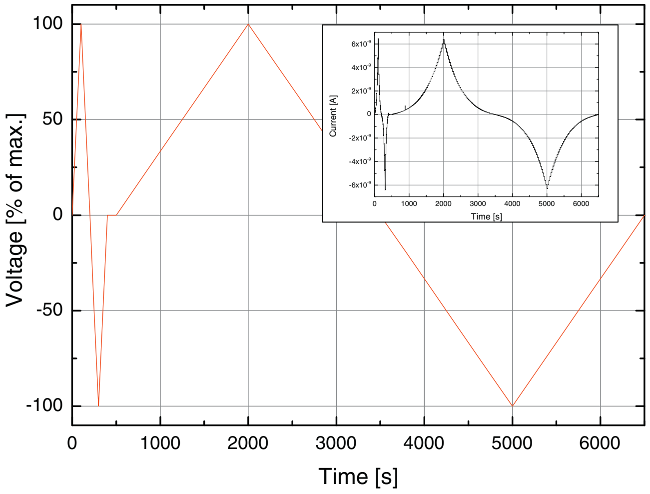

Fig. 1. The applied voltage sweep for the leakage current measurements. The inset shows the corresponding current response for a 100 nm thick SiN_x film deposited at 450 W/3 μbar.

>50 Ω cm, a thickness of 350 ± 15 μm and a diameter of 100 mm. Prior to the deposition the wafers were cleaned in acetone and isopropyl alcohol. The native oxide was removed using a short dip in buffered hydrofluoric acid (BHF), followed by rinsing in de-ionized (DI) water. Finally, the silicon surface was treated using an inverted sputter etcher (ISE) at a back pressure of 6 μbar and a power of 200 W in a pure argon atmosphere for 60 s.

The inverted sputter etching and the SiN_x deposition was done in a *Von Ardenne LS730S* sputtering system. The latter process was performed in a pure nitrogen atmosphere using DC magnetron sputtering in power control mode. A 150 mm diameter 6 mm thick polycrystalline silicon disc bonded to a copper plate served as target. Prior to the deposition the vacuum chamber was evacuated to a base pressure of $<10^{-7}$ mbar. The flow rate of the process gas N_2 was set to 60 sccm. The power of the DC plasma was 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 of ~390–410 V), and the back pressure during deposition between 3 and 9 μbar. The target–substrate distance was fixed to 65 mm. In the first screening experiments to determine the deposition rates at different sputter conditions, samples with an approximate thickness of 500 nm were produced by adjusting the deposition times.

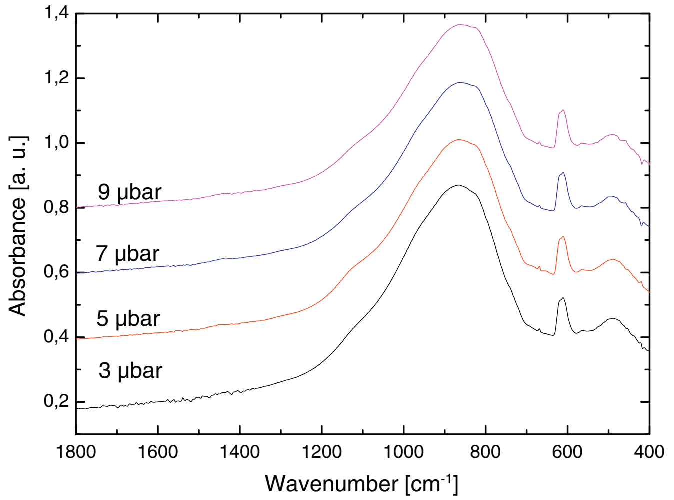

Fig. 3. FT-IR spectra of DC magnetron sputtered SiN_x thin films (deposited at 450 W DC power).

The thickness values and refractive indices of the samples were determined via the spectral reflectometry method using a *Filmetrix F20* system, by measuring the broadband reflectance spectrum between the wavelengths of 200 and 1100 nm. The biaxial stress of the thin films was calculated using Stoney's formula [27] after measuring the wafer bow prior to and after the deposition using an *EH metrology MX203* capacitive wafer geometry gauge. Fourier-Transform Infrared spectroscopy (FT-IR) measurements were taken using a *Bruker Tensor 27* infrared spectroscope using a resolution of 4 cm^{-1} between wavenumbers of 400 and 4000 cm^{-1}. For the determination of the mass density, the coated wafers were weighed using a laboratory scale prior to and after the deposition.

Etch resistance tests were performed in an *STS320* parallel plate RIE reactor, using a CF_4:O_2 gas mixture using the parameters from [28], in a *Xactix e1* Xenon difluoride plasmaless etching system (at an etch pressure of 4 mTorr), and by wet etching in a 1:7 mixture of 49% aqueous HF and DI water. For the different etch media, different sets of etch times were used, dependent on the expected etch rates: 2 and 4 min for RIE; between 1 and 30 min for HF, and 20 and 40 cycles of 30 s for XeF_2 etching. Determination of the etch depth was done with the spectral reflectance method as described above by measuring the change in film thickness.

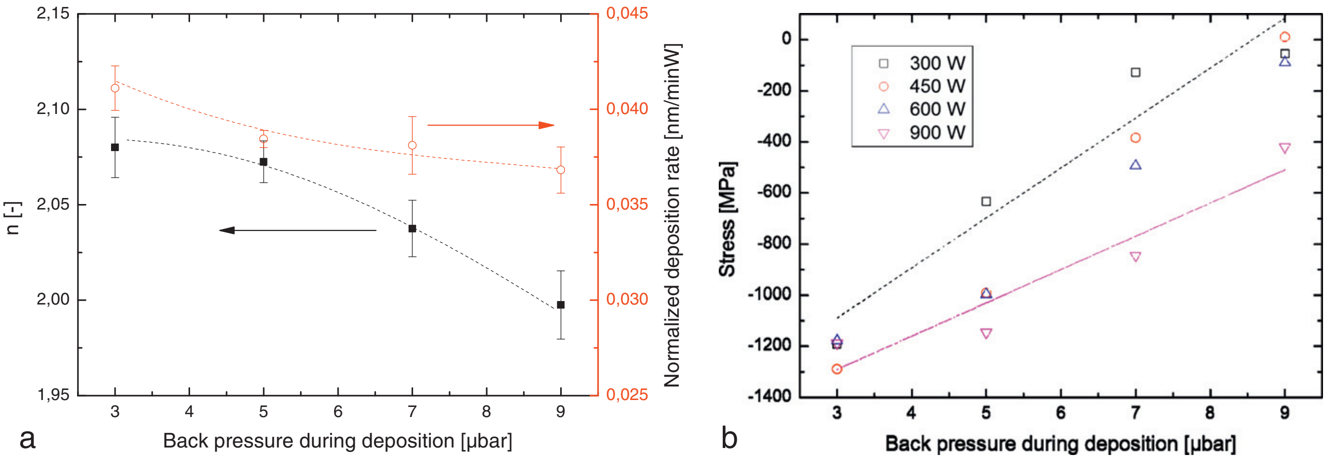

Fig. 2. Refractive indices and deposition rates normalized to unit power of the SiN_x thin film samples averaged over all DC power levels as a function of back pressure during the DC sputter deposition (a). Biaxial film stress of DC sputter deposited SiN_x thin films having a thickness of ~600 nm as a function of back pressure during deposition and sputter power (b). The inserted lines serve only as guides to the eye.