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A comparative micro-cantilever study of the mechanical behavior of silicon based passivation films

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ABSTRACT

A comprehensive study on the mechanical behavior of plasma enhanced chemical vapor deposited silicon oxide, oxynitride and nitride thin films is provided. Hardness, Young's modulus, yield stress, fracture stress and fracture toughness values are determined by the nanoindentation and the micro-cantilever deflection technique. The micro-cantilever deflection technique is discussed in terms of measurement accuracy and reproducibility and the results are compared with standard nanoindentation measurements. Correlations between the yield and fracture behavior, which have been observed for glass fibers, are discussed in this paper for dielectric thin film glasses.

1. Introduction

In microelectronic industry, plasma enhanced chemical vapour deposition (PECVD) materials are of common use as inter-layer-dielectric and chip passivation films [1]. Thermo-mechanical stresses [2], which are caused by the mismatch of the coefficients of thermal expansion between the dielectric film and the metal-lines in the chip, may introduce cracks and subsequent device failure. In recent years, the semiconductor industry started to employ mechanical simulations, which are based on finite element methods, to predict the life-time of their components [3]. To run the simulations material properties must be known at small length scales. For the silicon based dielectric thin film materials the tensile behavior is important as the materials fail under tension. However, these parameters are experimentally difficult to access. In the 1960s Marsh [4,5] studied the fracture behavior of macroscopic flaw-free glass fibers and correlated the yield stress, which was determined by a simple hardness measurement with the fiber strength under tension. Nowadays, such a correlation between the yield behavior under compression and the fracture behavior under tension could simplify mechanical robustness estimations as one could benefit from the simplicity and rapidness of a nanoindentation hardness measurement. In this paper it will be discussed whether the correlation reported by Marsh can also be observed for dielectric thin film glasses with thicknesses of ~800 nm.

In order to study the mechanical behavior of thin film materials in small dimensions, miniaturized mechanical test-methods are required, which pose a challenge to the applied techniques. While there are already some micro-scale mechanical testing methods available, huge efforts are still undertaken to improve existing and develop new methods for reliable mechanical measurements in micron and sub-micron dimensions. The micro-scale tension test [6,7], the wire bending test [8,9], the bulge test [2], nanoindentation [10–13], micro-compression [14], substrate curvature technique and the micro-beam deflection technique [15–18] are already existing techniques and are further improved. While nanoindentation and substrate curvature tests are not performed on freestanding films, the other methods, listed above, are. Reviews of micro-scale mechanical testing methods are given in [19–21].

In this paper, additionally to the well established nanoindentation technique, micro-cantilever deflection tests are performed to determine the fracture stress, the Young's modulus as well as the fracture toughness of different dielectric films.

A simple technique to investigate the fracture toughness of thin films on substrates is the nanoindentation technique, where a sharp indenter causes radial cracks in brittle materials [11–13]. The length of these radial cracks can be used to estimate the fracture toughness. In

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case of thin films on substrates, huge indents may be necessary to introduce detectable cracks. Unfortunately, in most cases there is a strong interaction between film and substrate which is hard to quantify. Thus this method can only deliver an estimate of the fracture toughness. Hence, the micro-cantilever deflection method, which was introduced by Di Maio and Roberts [22], is used to deduce the toughness more accurately.

All mechanical tests are performed on amorphous silicon oxide (a-SiO_xH_z), oxynitride (a-$SiO_xN_yH_z$) and nitride (a-SiN_yH_z) thin passivation films with different nitrogen contents in order to study the influence of the composition on the mechanical behavior systematically. The capabilities of the micro-cantilever deflection technique are shown and the results are discussed.

2. Experimental details

2.1. Materials

Amorphous PECVD silicon oxide (a-SiO_xH_z), amorphous PECVD silicon oxynitride (a-$SiO_xN_yH_z$) and amorphous PECVD silicon nitride (a-SiN_yH_z) thin films are prepared from gas mixtures of silane (SiH_4), nitrogen (N_2), nitrous oxide (N_2O) and ammonia (NH_3) using radio frequency assisted deposition at elevated temperature. All films are prepared on 8 inch (200 mm diameter) (100) silicon wafers using a industrial production equipment.

Thin film composition is determined by Rutherford backscattering spectroscopy (RBS) and elastic recoil detection (ERD) [23,24] using ^{4}He-ions with energies of 2.4 MeV and 2.7 MeV for RBS and ERD, respectively. The scattering angles are 30° and 170° for RBS and ERD, respectively. Additionally, fourier transformation infrared spectroscopy (FTIR) is performed using a Biorad QS 2200 equipment which is able to scan over a wavenumber range from 400 cm^{-1} to 4000 cm^{-1}. The FTIR absorption spectra of silicon oxide, silicon oxynitride and nitride are illustrated in Fig. 1. The absorption bands and their corresponding vibrations are inserted in the figure. The details about their kind of vibrations can be found elsewhere [1,25–29].

To detect possible compositional changes when depositing thin films of different thicknesses, the refractive index of the dielectric materials was measured. To determine the thickness and the refractive index, a refractometer (Opti-Probe 6420 system), with a wavelength of 675 nm is used. The RBS, ERD and refractive index of the dielectric films analyzed are summarized in Table 1.

X-ray diffraction (XRD) θ–2θ measurements with diffraction angles between 2θ = 5° and 50° were performed using CuK_α radiation

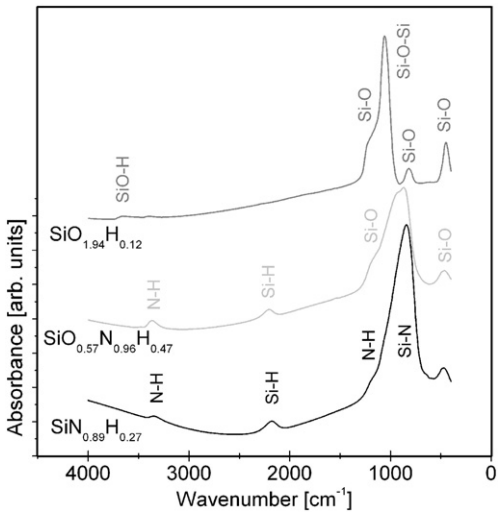

Fig. 1. FTIR absorption spectra of silicon oxide, oxynitride and nitride. The assigned bonds are provided in the figure.

Table 1
Chemical composition of the materials investigated, determined by RBS and ERD measurements.

Material Name	Refractive Index	O/Si	N/Si	H/Si
Silicon oxide	1.46	1.94	0.00	0.12
Silicon oxynitride	1.72	0.57	0.96	0.47
Silicon oxynitride	1.76	0.37	1.01	0.43
Silicon nitride	1.91	0.00	1.29	0.30
Silicon nitride	2.1	0.00	0.89	0.27

to detect crystalline inclusions in the amorphous materials. A Bragg-Brentano geometry (Seifert PTS 3000) was used with a step size of 0.1° and 2 s time per step. XRD θ–2θ measurements reveal that silicon oxide, oxynitride and nitride films are amorphous without any crystalline phases.

To investigate defects at nanometer scale, which can influence the strength of the materials, grazing incidence small angle scattering (GISAXS) was performed. A Bruker Nanostar equipment (sealed X-ray tube, 1.5 kW, focal spot = 0.4 mm × 0.8 mm, Cu-K_α, 40 kV/35 mA, pair of crossed Göbel mirrors, Bruker AXS Hi-STAR position sensitive area detector) was employed. GISAXS at incidence angle of ~0.2° and an integration time of 16 h on both, silicon substrate and thin film on substrate, reveal that there is almost no difference in the scattered signal of the two systems. One can conclude that there are either no electron density differences at the nanometer scale or the electron density differences are too small to be detected.

Film stresses, which can influence the hardness measured by nanoindentation, are determined using the laser scanning substrate curvature technique. These stresses result from differences in thermal expansion coefficients of film and substrate. Substrate curvature is measured before and after oxide growth with a FSM 128 laser scanning equipment. As the film thickness is negligibly small (less than 2% of the substrate thickness (725 μm)) the Stoney equation can be applied to calculate the stress in the thin film, σ, from the curvature, *R*, with:

$$\sigma = \frac{M_s t_s^2}{6 t_f} \frac{1}{R} \tag{1}$$

$M_s = E/(1-\nu)$ denotes the biaxial modulus of the (100) silicon substrate material, ν the Poisson ratio of the substrate material, t_s the substrate thickness and t_f the film thickness. Refractive index and film stress were measured 6 weeks after film deposition again. No change in refractive index was observed, indicating that there is no detectable compositional change by out-gassing hydrogen and no change of the residual stress by viscous flow. Hence, it is assumed that the mechanical properties remain stable in this time range.

The immediate result of an indentation experiment is the hardness and the reduced modulus, that takes into account the deformation of the indenter tip and the lateral elastic deformation of the material via its Poisson's ratio [10].

Reduced modulus, E_R, and hardness, *H*, of film and substrate are measured with a Hysitron Triboscope mounted on the scanner head of a Digital Instruments 3100 atomic force microscope using a Berkovich indenter. Hardness measurements are conducted on ~2 μm thick films on (100) silicon substrate with maximum penetration depths of 10% of film thickness to exclude substrate-effects. Determination of area function and data evaluation is performed according to [10].

The hardness is calculated from the contact area of the indenter, *A*, and the maximum load applied, F_{max}, via the relation:

$$H = \frac{F_{max}}{A}. \tag{2}$$

The reduced modulus, E_R, is defined in Eq. (3) where *E* and ν are the Young's modulus and Poisson's ratio of the thin film on silicon