Influence of process parameters on the properties of TEOS DF-PECVD grown SiO_x films by DOE

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The aim of this work is to optimize deposition process for thin silicon dioxide films preparation and to study the influence of stress in these films. SiO_x films have been deposited by DF-PECVD (dual frequency plasma enhanced chemical vapor deposition) method in the Applied Materials P5000 single wafer tool. The main purpose of using the high frequency is to generate reactive species and to provide sufficient electron and ion densities. Low frequency is used to control plasma sheath voltage (by controlling the ion bombardment it is possible to eliminate intrinsic stress) [2]. This reactor is a multichamber vacuum load lock deposition system. DF-PECVD (13.56 MHz and 350 kHz) process uses a mixture of TEOS (tetraethoxysilane) and O₂ as a reactive gas to deposit thin SiO_x films. For better uniformity of films, the upper electrode had a showerhead design.

Silicon wafers (150 mm, 525 mm thickness, (100) orientation, n-type, borone doped) have been used as a substrate. Susceptor temperature has been held on the constant temperature of 400°C (\sim 357°C on silicon wafer) by the use of infrared (IR) lamps. Chamber pressure during deposition process was 4.8 torr and temperature of bubbler 39 °C. The power of high and low frequency, inter-electrode spacing and gas flow of TEOS has been changed.

Deposition rate, etch rate, uniformity and refractive index have been measured by ellipsometry (Rudolph FE VII) in 9 points on the wafer. Non-uniformity values were calculated using (standard deviation/mean) x 100. Etch rate has been acquired using the timed buffered oxide etch (1 min., BOE 7:1). The residual stress has been measured by KLA-Tencor FLX-2320. The stress in thin film has been calculated from the radius of curvature of the substrate. In general, for thin film processes, residual stress is the sum of thermal and intrinsic stresses. Due to the difference in thermal -- mechanical properties between the film and substrate, thermal stress is usually unavoidable. Intrinsic stress is generated during the film growth and is strongly dependent on process conditions. Attractive forces within pores can lead to tensile stress, while gas entrapment and its physi- or chemisorption in the inner cavities or at grain boundaries may lead to compressive stress [1]. SiO_x films prepared by PECVD technique are always in compressive stress but after deposition process the films are annealed and it is highly probable that compressive stress will change to tensile stress. While compressive stresses lead to wrinkling and film delamination, tensile stresses lead to film fracture [3]. The dependence of fracture in thin films on residual stress and annealing process (60 min, 1000 °C, in vacuum and in the air) has been studied by SEM (Scanning electron microscopy) JEOL JSM 6700F. Figure 1 shows fractures in 1µm and 3µm films after annealing process in vacuum.

The influence of power of low frequency, high frequency, inter-electrode spacing and gas flow of TEOS on deposition rate, etch rate, refractive index, uniformity and stress have been studied by DOE (Design of experiment). Full factorial (two levels) Design of experiment has been designed and evaluated in statistical software JMP 7 (Figure 2).

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Figure 1. Fractures in thin SiO_2 film after annealing (60 min, 1000°C, in vacuum). In 1µm film cracks were observed localy on wafer surface while in 3µm film cracks were nearly on the hole wafer surface.



Figure 2. Design of experiment and final table. The influence of power of low frequency (LF), high frequency (HF), inter-electrode spacing (Spacing) and gas flow of TEOS (TEOS) on deposition rate (DR), etch rate (ER), refractive index (RI), non-uniformity and stress.