2000

Structural Properties and Stress Analysis of SiO2 Thin Films

G. Joyce
Trinity College Dublin

C. Moore
Trinity College Dublin

T. S. Perova
Trinity College Dublin

See next page for additional authors

Structural Properties and Stress Analysis of SiO_2 thin films

G. Joyce[1], C. Moore[1], T.S. Perova[1], C. Beitia[2], A. Kocot[3], K. Berwick[4], R.A. Moore[1] H. Byrne[4] and J.F. McGilp[2]

[1]*Department of Electronics & Electrical Engineering, University of Dublin, Trinity College, Dublin 2, Ireland.*
[2]*Department of Physics, University of Dublin, Trinity College, Dublin 2, Ireland.*
[3]*Institute of Physics, Silesian University, Katowice, Poland.*
[4]*Department of Electronics & Communications Engineering, Dublin Institute of Technology, Kevin Street, Dublin 8.*

Abstract

Wet and dry oxidations of (111) crystalline silicon wafers were performed at temperatures between 900°C and 1150°C and subsequently annealed at 700°C. Process parameters such as the withdrawal rate from the furnace and annealing time were varied and the effect on the oxide layer are discussed. The magnitude and nature of the stress in the wafers after thermal oxidation was analysed using micro-Raman spectroscopy. The Full Width at Half Maximum (FWHM) of the Raman peak at ~ 520 cm^{-1} was also analysed to extract information relating to crystal disorder and defect density caused by the oxidation process. The results obtained from the Raman technique are compared with FTIR results and theoretical predictions from bow measurements using surface profilometry. It was found that the stress is lower if the wafer is cooled slowly in the furnace tube. The addition of chlorine to the ambient during oxide growth, or increasing the annealing time, reduces the wafer bow and the consequent stress.

FTIR and ellipsometry data indicate that the wafer oxidised at 900°C shows the highest density and refractive index. These results are consistent with results obtained recently for (100) silicon wafers [1]. The densification process exhibits two distinct temperature regimes, viz. a low-temperature regime and a high temperature regime, in accordance with the atomic model predictions described in Ref. [2].

---

1. Introduction

Manufacture of a silicon integrated circuit is a complex procedure incorporating many different processes and a wide variety of materials. Thermal cycling of the chip and thermal mismatch between deposited layers can lead to the development of stress within a device, ultimately manifesting itself in device failure.

Stress at the Si-SiO_2 interface occurs due to thermal mismatch between the two materials and causes the wafer to bow. This bowing is undesirable as it causes complications at a crystalline level because of the creation of defects. It also causes problems with photolithography due to the difficulty of getting a sharp focus across a distorted wafer.

In this work, we present the results of investigations of the stress developed within oxidised silicon wafers and the structural properties of thermally grown SiO_2 layers. The influence of the growth temperature and ambient composition on the oxide layer are also explored. A variety of assessment tools, including micro-Raman spectroscopy, FTIR spectroscopy, spectroscopic ellipsometry and surface profilometry were used in this study.

2. Sample preparation

100mm diameter, n-type, (111) Czrochralski grown silicon wafers with 3-5 Ωcm resistivity were used throughout this study. Wet and dry oxidations were performed at different temperatures and various process parameters, including the withdrawal rate from furnace and the anneal time, were varied and the impact of this on the oxide was investigated.

3. Experimental

Surface profilometry

The bow of each wafer was measured before and after oxidation on a vibration free table using a Talysurf© profilometer. Goklaney et al. reported that if w is the maximum deflection of a wafer at the centre, E is the elasticity (Young's) modulus for silicon, h is the thickness of the wafer, v_{Si} is Poisson's ratio for silicon and r is the radius of the wafer then Eqn. (1) may be used to allow the stress σ_0, to be calculated.

$$\sigma_0 = \frac{3Eh^2 w}{r^3(1-v_{Si})(4+v_{Si})} \tag{1}$$

Micro-Raman spectroscopy

Raman spectroscopy is the measurement of the wavelength and intensity of inelastically scattered light[3]. The technique is sensitive to stress within silicon. A Dilor Labram ISA Raman spectrometer was used for all measurements. The Raman shift is substituted into Eqn. (2) below to yield a quantitative value for the uniaxial stress [3].

$$\sigma = -\frac{\Delta\omega}{2x10^{-9}} \tag{2}$$

where $\Delta\omega$ is the difference between the measured peak of the stressed silicon and the peak of the unstressed reference silicon. σ is the stress measured in Pascals.

FTIR spectroscopy

A Digilab FTS60A FT-IR spectrometer was used to carry out the IR measurements on the all samples. Experiments were carried out in order to look at (i) the measurement of the oxide thickness using IR techniques and its comparison to ellipsometry measurements, (ii) the density of the oxide layer with decreasing temperature, and (iii) the vibrational fingerprint of the Si/SiO_2 system with the IR light at normal incidence and at an angle to the sample surface.

Results and Discussion

Surface profilometry and Raman measurements

A direct comparison between surface profilometry measurements and Raman spectroscopy is made for both wet oxides and dry oxides. The effects of withdrawal rate from the furnace, anneal time and the addition of chlorine to the process gas on the stress was also explored. Representative results for the wet oxide are shown below in Figure 1.

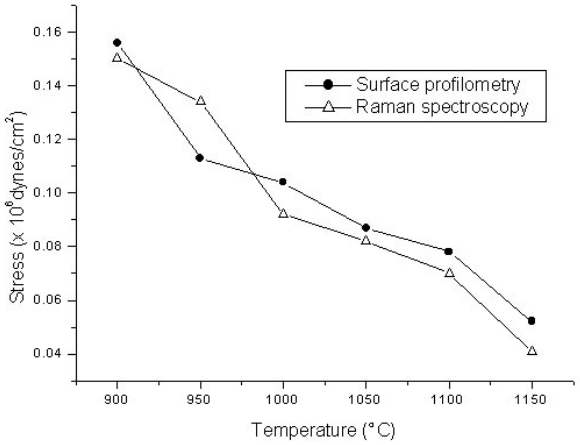

Figure 1. Wet oxide stress measurements

Both the surface profilometry and the Raman spectroscopy measurements are in good agreement. Clearly, the stress at the Si/SiO_2 interface decreases with increasing oxide growth temperature. Further measurements indicate that this trend is observed regardless of whether a dry or wet oxidation process is performed. We also find that the bow decreases as the anneal time increases or if TCE is added to the process gas.

FTIR measurements

Figure 2 shows how the peak position and intensity of the $1070cm^{-1}$ (TO) band changes with oxidation temperature. The peak position shifts to the high frequency side up to 1050°C and then abruptly decreases. Shift in this IR absorption band may be related to the physical variation of the internal structure of the oxide. From data on plastically densified SiO_2 it has been found [4] that the TO mode frequency shift ,*dv*, with density *dp* is as defined in Eqn. (3).

$$\frac{dv}{dp} = -93 cm^2 g^{-1} \tag{3}$$

It can be estimated that for the 900°C sample the absorption peak is shifted by ~$2.9cm^{-1}$ with respect to the 1050°C sample. From equation (3) it can be deduced that the 900°C oxide has a density ~ 1.14% larger than that of the 1050°C oxide.

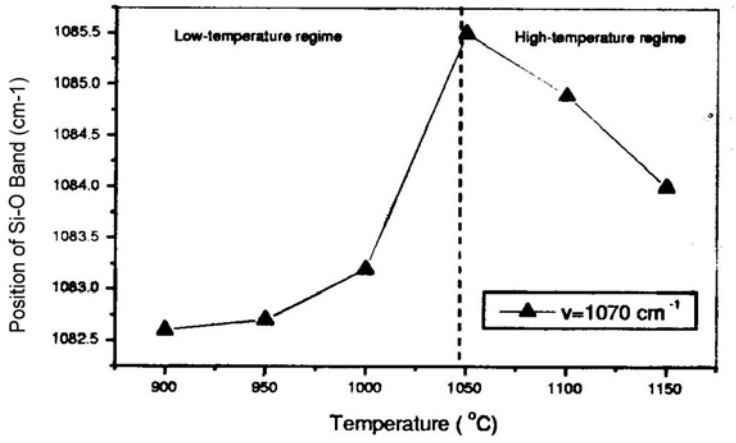

Figure 2. Peak position of the $1070cm^{-1}$ band versus temperature

Analysis of the intensity of the TO (1070cm^{-1}) band using a calibration plot reported in [5] allows an evaluation of the oxide thickness. In this study, the greatest thickness was recorded for the 1050°C sample (0.29μm), while for 900°C and 1150°C samples the thickness was 0.26μm and 0.24μm respectively. These results are largely consistent with ellipsometry measurements.

5. Conclusions

Surface profilometry, Raman spectroscopy and FTIR methods have been used to characterise stress at the Si-SiO_2 interface. The results obtained from the surface profiles agreed with those obtained with the Raman method. We conclude that the lower the oxidation temperature, the greater the stress on the wafer. Increasing the annealing time or the addition of chlorine in the form of TCE reduces wafer stress considerably.
A study of the Si-O-Si bond was conducted using Infra-Red spectroscopy. It was observed that the density of the SiO_2 layer changed with oxidation temperature. This is attributed to the change in the molecular structure of the Si-O-Si bonds within the oxide. A comparison of the IR technique to ellipsometry confirmed the change in density of the oxide as a function of oxidation temperature.

References

[1] C. Martinet and R.A.B. Devine, *J.Appl.Phys.*, **77**, 4343 (1995).
[2]. G. Lucovsky, M.J. Manitini, J.K. Srivastava, & E.A. Irene, J. *Vac. Sci. Technol.*, Vol. B5 (2), pp. 530-537, 1986.
[3]. I. De Wolf, *J. Sci. Technol*., Vol. 11, pp. 139-154, 1996.
[4]. R.A.B. Devine, *Trans. Mater. Res. Soc*., 8, pp. 165, 1992.
[5]. I.W. Boyd & J.J.B. Wilson, *J. Appl. Phys.*, Vol. 53, pp. 4166, 1982.
[6]. S.M. Goklaney, W.A. Porter, & T.J. Kozik, *Indian J. Tech*., Vol. 13, 1975.