Non-destructive Characterization of Nitrogen-implanted Silicon-on-insulator Structures by Spectroscopic Ellipsometry*

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Abstract

Silicon-on-insulator (SOI) structures implanted with 200 or 400 keV N⁺ ions at a dose of $7.5 \times 10^{17} \text{ cm}^{-2}$ were studied by spectroscopic ellipsometry (SE). The SE measurements were carried out in the 300–700 nm wavelength (4.13–1.78 eV photon energy) range. The SE data were analysed by the conventional method of using appropriate optical models and linear regression analysis. We applied a seven-layer model (a surface oxide layer, a thick silicon layer, upper two interface layers, a thick nitride layer and lower two interface layers) with good results. The fitted parameters were the layer thicknesses and compositions. The results were compared with data obtained from Rutherford backscattering spectroscopy (RBS) and transmission electron microscopy. The sensitivity of our optical model and fitting technique was good enough to distinguish between the silicon-rich transition layers near the upper and lower interfaces of the nitride layer, which are unresolvable in RBS measurements.

1. Introduction

High dose implantation of reactive ions, such as oxygen, nitrogen or both, into silicon wafers has been successfully used to synthesize silicon-on-insulator (SOI) structures suitable for very-large-scale integration and radiation hard applications [1, 2]. The advantages of SOI devices have been noted by many investigators [3–5]. Parameters affecting the SOI microstructures have been extensively studied by Rutherford backscattering spectroscopy (RBS), transmission electron microscopy (TEM) imaging, secondary ion mass spectrometry and Auger electron spectroscopy [6–10]. Quite recently, spectroscopic ellipsometry (SE) has been used with success in the investigation of oxygen-implanted SOI [11, 12]. However, nitrogen-implanted SOI is more difficult, because of the stronger dependence of the microstructure on implantation dose.

In this paper, we report the results of the characterization of SOI structures formed by a buried Si₃N₄ layer. It is worth noting that the investigated samples are probably not the best for technological use. The emphasis in this paper is placed on the characterization method which may be the tool for future optimization experiments.

In a previous paper [13], we applied multiple-angle-of-incidence (single-wavelength) ellipso-

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metry (MAIE) to determine the refractive indices and thicknesses of the same SOI layers using a relatively simple three-layer optical model. The results reported in that paper are shown together with the present results in Tables 3 and 4.

2. Experimental details

Nitrogen ions were implanted at 200 keV (sample 1) and 400 keV (sample 2) into $\langle 100 \rangle$ p-type ($12 \Omega \text{ cm}$) single-crystal silicon using the 500 keV ion implanter at the Central Research Institute for Physics, Budapest. The samples were given a dose of $7.5 \times 10^{17} \text{ N}^+ \text{ cm}^{-2}$ with a current density of $2 \mu\text{A cm}^{-2}$ on a $2 \text{ cm} \times 2 \text{ cm}$ surface. External target heating during implantation was applied at 600°C . Post-implantation annealing was performed at 1300°C for 2 h in argon for sample 1 and at 1200°C for 2 h in N_2 for sample 2. The silicon surface was covered with another piece of silicon in order to protect the sample surface from oxidation during high temperature annealing (face-to-face arrangement).

RBS combined with channelling was performed with 2 MeV He^+ ions at a scattering angle of 165° .

The SE measurements were performed on a rotating analyser ellipsometer in the 300–700 nm wavelength (4.13–1.78 eV photon energy) region at the Twente Technical University [14].

The cross-sectional TEM investigation was made at the University of Oxford.

3. Model variables

Data were analysed using a FORTRAN program which calculates $\tan \psi$ and $\cos \Delta$ values for an assumed input optical model of the sample and then minimizes the mean-square difference between measured and calculated $\tan \psi$ and $\cos \Delta$ values by varying the designated model parameters according to the Fletcher–Powell [15] algorithm. With reference to the work of Aspnes *et al.* [16], the quantity used to describe the agreement between the experimental data and the modelling process is defined as the unbiased estimator σ . For the analysis of the samples the following expression was used:

$$\sigma = \left[\frac{1}{n - p - 1} \times \left\{ \sum_{j=1}^n (\cos \Delta_j^{\text{exp}} - \cos \Delta_j^{\text{calc}})^2 + (\tan \psi_j^{\text{exp}} - \tan \psi_j^{\text{calc}})^2 \right\} \right]^{1/2}$$

where n is the number of experimental measurements and p is the number of unknown model parameters. The superscripts exp and calc refer to the experimental and calculated values respectively.

We used three basic models (Fig. 1). The first (Fig. 1(a)) consists of a silicon substrate, a nitride (mixed with silicon) layer, a silicon (mixed with

Fig. 1. Optical models used for SE data evaluation. The t_i values are the layer thicknesses, and the v_i values are the volume fractions of the first components in each layer. (Thus the $1 - v_i$ values are the volume fractions of the second components.)

nitride) layer and a surface oxide (mixed with silicon) layer. The second (Fig. 1(b)) and the third (Fig. 1(c)) models are enhanced with two and four additional interface layers respectively. Each layer is modelled using the Bruggeman effective-medium model with two end-member complex dielectric functions (or complex refractive index functions) of crystalline silicon (c-Si) [17] or SiO_2 [18] or Si_3N_4 [19]. These functions are not necessarily the most appropriate for describing these layers but are the best obtainable from literature. However, they are good starting points. Each layer is described by the volume fractions of the constituents. If the volume fraction is less than zero or greater than unity, this means that the actual refractive index of the layer is less or greater than the basic value of unity.

During the fitting process, not all the parameters were varied. Firstly, we investigated only the surface layer considering only the 300–400 nm wavelength region. After having

fitted the parameters (the thickness and volume fraction) of the surface layer, we fitted the other parameters using the whole wavelength region, but now having fixed the parameters of the surface. The imaginary part of the complex refractive index of single-crystal silicon (which is proportional to the light absorption) is greater than 0.4 in this (high photon energy) region, meaning that the optical penetration depth is less than $0.2 \mu\text{m}$. This is supported by the presented simulations (Fig. 2). There is no difference in the curves up to 400 nm.

The rapidly changing interference pattern is due to the silicon layer which is sandwiched between the surface film and the buried nitride layer. The structure of this pattern (the number and places of the oscillations, and the amplitudes) is very sensitive to the thicknesses.

In c-Si the optical penetration depth of light depends on its wavelength. In general, the higher the wavelength, the deeper is the penetration. In

Fig. 2. Simulated SE spectra on the inserted optical models: (a) 20 nm variation in the silicon; (b) 20 nm variation in the nitride.

other words the measurements at longer wavelengths contain more information in the buried layer. This fact is shown in Fig. 2(b). If the thickness of the buried nitride layer is changed by 20 nm, there is a great variation mainly above 500 nm, the difference increasing with increasing wavelength.

The main problem was the choice of the initial parameters. We must make a good guess for the thicknesses as otherwise the program can "miss" or, in other words, can find a false minimum. We used computer simulations similar to that in Fig. 2.

4. Results and discussion

The best fits are presented in Figs. 3 and 4. The first model (Fig. 1(a)) cannot satisfactorily describe the samples, although the thicknesses can be determined with great precision. After having completed the models with interface

layers (Figs. 1(b) and 1(c)), the fittings became increasingly better. The unbiased estimator σ decreased by more than a magnitude. (We did not study the effect of more interface layers. More layers mean more parameters—each additional layer gives two more parameters and thus increases the computing time. The most sophisticated model took more than 5 h on our IBM PC/AT computer and the time increased as the square of the number of parameters.)

The materials parameters which yielded the best-fit spectra to the experimental data are shown in Tables 1 and 2. The most interesting results are the silicon-rich thin transition layers near the upper and lower interfaces of the nitride layer. This result is comparable with the Auger depth profile measurement of Oosting *et al.* [10] on a sample (160 keV, $7 \times 10^{17} \text{ N}^+ \text{ cm}^{-2}$; 1200 °C for 1 h) similar to sample 1.

The volume fraction values of these layers near to or less than zero can be interpreted in the fol-

Fig. 3. Plot of $\cos \Delta$ and $\tan \psi$ vs. wavelength of sample 1 fitted by the optical models in Fig. 1.

Fig. 4. Plot of $\cos \Delta$ and $\tan \psi$ vs. wavelength of sample 2 fitted by the optical models in Fig. 1.

TABLE 1 Best fits on sample 1

Model 1, $\sigma = 0.048$	Model 2, $\sigma = 0.008$	Model 3, $\sigma = 0.001$
$t_2 = 351 \text{ nm}, v_2 = 1.02$	$t_2 = 352 \text{ nm}, v_2 = 1.00$	$t_2 = 352 \text{ nm}, v_2 = 0.99$
$t_3 = 175 \text{ nm}, v_3 = 0.96$	$t_3 = 35 \text{ nm}, v_3 = 0.41$ $t_4 = 130 \text{ nm}, v_4 = 0.94$ $t_5 = 18 \text{ nm}, v_5 = 0.05$	$t_3 = 27 \text{ nm}, v_3 = 0.66$ $t_4 = 6 \text{ nm}, v_4 = -0.25$ $t_5 = 130 \text{ nm}, v_5 = 0.93$ $t_6 = 7 \text{ nm}, v_6 = -0.18$ $t_7 = 12 \text{ nm}, v_7 = 0.67$

TABLE 2 Best fits on sample 2

Model 1, $\sigma = 0.0032$	Model 2, $\sigma = 0.0016$	Model 3, $\sigma = 0.00025$
$t_2 = 601 \text{ nm}, v_2 = 0.99$	$t_2 = 601 \text{ nm}, v_2 = 0.98$	$t_2 = 601 \text{ nm}, v_2 = 1.00$
$t_3 = 255 \text{ nm}, v_3 = 0.75$	$t_3 = 36 \text{ nm}, v_3 = 0.50$ $t_4 = 200 \text{ nm}, v_4 = 0.73$ $t_5 = 24 \text{ nm}, v_5 = 0.34$	$t_3 = 29 \text{ nm}, v_3 = 0.71$ $t_4 = 10 \text{ nm}, v_4 = 0.31$ $t_5 = 200 \text{ nm}, v_5 = 0.73$ $t_6 = 9 \text{ nm}, v_6 = 0.14$ $t_7 = 18 \text{ nm}, v_7 = 0.56$

lowing manner. We use the dielectric function of single-crystal silicon. However, these layers probably consist of small damaged silicon crystallites; so their dielectric functions tend towards that of amorphous silicon. Thus the use of the dielectric function of c-Si results in a volume fraction greater than the real value.

The volume fraction of the thick nitride layer is a very useful parameter. It can show the quality of the buried insulator layer. For sample 2 the implanted dose is obviously too small. This is because the peak concentration of the as-implanted nitrogen profile is lower than that for sample 1 because of the higher range straggling (higher implantation energy).

The results were compared with data from other independent measurements. In Figs. 5 and 6 the results from RBS and TEM are presented. We can conclude that the layer thickness data are in very good agreement (Tables 3 and 4). Moreover, the TEM pictures show that the upper nitride-silicon interface is rougher and thicker than the lower interface.

Fig. 5. RBS channelling spectra of (a) sample 1 and (b) sample 2.

Fig. 6. Cross-sectional TEM pictures on sample 1. In the surface single-crystal silicon layer, three dislocations can be seen.

TABLE 3 Collected data of sample 1. (multiple-angle-of-incidence ellipsometry results are from ref. 13)

Parameter	Value obtained by the following techniques		
	MAIE [13]	RBS (nm)	TEM (nm)
Thickness of top silicon (nm)	355	350	380
Thickness of buried nitride (nm)	177		180
Complex refractive index of top silicon	3.89-0.04i		
Complex refractive index of nitride	2.03-0.00i		

TABLE 4 Collected data of sample 2. (multiple-angle-of-incidence ellipsometry results are from ref. 13)

Parameter	Value obtained by the following techniques	
	MAIE [13]	RBS
Thickness of top silicon (nm)	603	600
Thickness of buried nitride (nm)	250	
Complex refractive index of top silicon	3.89-0.02i	
Complex refractive index of nitride	2.57-0.00i	

5. Conclusion

It was demonstrated that SE using linear regression analysis can give information not only about the thickness but also on the composition of each layer and interface of the SOI structure formed by thermally activated redistribution of high dose ion-implanted nitrogen. The agreement between SE and other analytical techniques (RBS and cross-sectional TEM) is fairly good.

The advantages of SE over conventional ellipsometry originate from the fact that it utilizes the varying optical refraction and absorption with photon energy to obtain information about the multilayer system under investigation.

It is worthwhile emphasizing that the considerable sensitivity of SE to the interface properties, layer thicknesses and microstructure can give it an essential role in future optimization experiments.

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References

- 1 H. W. Lam, R. F. Pinizzotto and A. F. Tasch, Jr., *VLSI Handbook*, Academic Press, New York, 1985, p. 503.
- 2 H. W. Lam, A. F. Tasch, Jr., and R. F. Pinizzotto, *VLSI Electronics: Microstructure Science*, Vol. 4, Academic Press, New York 1982.
- 3 K. Izumi, M. Doken and H. Ariyoshi, *Electron. Lett.*, **14** (1978) 593.
- 4 G. Zimmer and H. Vogt, *IEEE Trans. Electron. Devices*, **30** (1983) 1515.
- 5 S. L. Patridge, *IEEE Proc.*, **133** (1986) 66.
- 6 P. L. F. Hemment, R. F. Peart, M. F. Yao, K. G. Stephens, R. J. Chater, J. A. Kilner, D. Meekison, G. R. Booker and R. P. Arrowsmith, *Appl. Phys. Lett.*, **46** (1985) 952.
- 7 P.-H. Chang, C. Slawinski, B.-Y. Mao and H. Lam, *J. Appl. Phys.*, **61** (1987) 166.
- 8 W. Skorupa, K. Wollschlager, M. Woelskow, K.-H. Heinig and H. Bartsch, *Proc. European Materials Research Society Meet.*, Vol. XV, Les Editions de Physique, Paris, 1987, p. 629.
- 9 W. Skorupa, K. Wollschlager, U. Kreissig, R. Grötzschel and H. Bartsch, *Nucl. Instrum. Methods B*, **19-20** (1987) 285.
- 10 P. H. Oosting, J. Petruzello and T. F. McGee, *J. Appl. Phys.*, **62** (1987) 4118.
- 11 J. Narayan, S. Y. Kim, K. Vedam and R. Manukonda, *Appl. Phys. Lett.*, **51** (1987) 343.
- 12 Z. Liang and D. Mo, *Appl. Phys. Lett.*, **52** (1988) 1050.
- 13 N. Q. Khanh, M. Fried, G. Battistig, Z. Laczik, T. Lohner, E. Jároli, V. Schiller and J. Gyulai, *Physica Status Solidi A*, **108** (1988) K35.
- 14 T. Holtslag, *Ph.D. Thesis*, Twente Technical University, Enschede, 1986.
- 15 R. Fletcher and M. J. Powell, *Comput. J.*, **6** (1963) 163.
- 16 D. E. Aspnes, J. B. Theeten and F. Hottier, *Phys. Rev. B*, **20** (1979) 3292.
- 17 D. E. Aspnes and A. A. Studna, *Phys. Rev. B*, **27** (1983) 985.
- 18 H. R. Philipp, *J. Phys. Chem. Solids*, **32** (1971) 1935.
- 19 H. R. Philipp, *J. Electrochem. Soc.*, **120** (1973) 295.