

## Silicon surface modified by H<sup>+</sup> ion plasma immersion implantation and thermal oxidation

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In this contribution results on the study of modification of Si surface through shallow plasma immersion ion (PII) implantation of H<sup>+</sup> ions with energy of 2 keV and doses ranging from 10<sup>13</sup> H<sup>+</sup>/cm<sup>2</sup> to 10<sup>15</sup> H<sup>+</sup>/cm<sup>2</sup> are presented. The implantation was regarded to proceed into Si through native SiO<sub>2</sub> layer. The structure and the optical properties of thin hydrogenated Si layer are characterized using spectroscopic ellipsometry (SE) and simulation of the distributions of the ions and implantation induced defects. Two-layer optical models are applied for examination of the composition and dielectric function behavior of the formed structures. The native oxide is found to be 3 nm thick. The thickness of the Si modified layer ranged between 23 to 14 nm depending on ion fluence. After oxidation the formed SiO<sub>x</sub> layers were characterized by optical spectroscopy methods. Observed variations in oxide composition and optical parameters are connected with the structure of the PII modified Si layer being oxidized, leading to lower intrinsic oxide stress (below  $\sim 7 \times 10^8$  N/m<sup>2</sup>) and smaller oxide refractive index as compared to the stoichiometric SiO<sub>2</sub>. The built-in internal stress was evaluated from the displacements of the characteristic peaks at 3.4 eV of Si in the RS and ER spectra and from the refractive index gained by SE. The results indicate the possibility to obtain structures with reduced structural strains and low concentration of defects.

**Key words:** Hydrogen implantation, PIII Si Surface Modification, Stress

### INTRODUCTION

Modification of surfaces has gained increasing importance for device fabrication in the next generation when the active regions are located in shallow surface layers. Incorporation of hydrogen into silicon offers the possibility to build very thin nanosized silicon oxide layers that can have different role in silicon based structures and devices.

However, incorporation of hydrogen in silicon device can have detrimental as well beneficial effects on device parameters. The purpose is to enhance beneficial and surpass detrimental. For that reason the role of hydrogen has been the subject to intensive investigations in the past decade. Among different beneficial effects stabilization of device characteristics through gettering of impurities and saturation of defects for future nanoelectronic applications, for exfoliation of silicon in smart cut process, increase carrier life time in solar cells, etc., can be stated. The detrimental effect appear mostly because the passivated through hydrogen saturation of dangling Si bonds defects can act as weak Si-H states which have the tendency to disrupt and annihilate the passivation effect upon external influence and long device exploitation. Several

hydrogen related defects, i.e. Si-H bonds, hydrogen induced platelets, monatomic hydrogen, hydrogen molecules, and hydrogen-related complexes have been discussed in the literature [1,2]. The mechanisms that govern interactions of hydrogen-related species with silicon defects and their role in grown process are still under debate.

Future new developments require detailed studies of hydrogen behavior in semiconductors. Of special interest is the behavior of hydrogen on the silicon surface and its role in the oxide growth process. Hydrogen can easily be introduced into semiconductors through proper hydrogen containing environments. In Si technology hydrogenation is usually achieved by implantation process, either beam [1,3] or plasma ion implantation (PIII) [4,5]. In spite of the advantages (shallow implants and large implanted area) PIII technique still is related with formation of lattice defects induced by collisions of implanted ions with the surface and atoms of substrate material in addition to plasma radiation.

The present study is devoted to the mechanism of influence of hydrogen introduction in Si through ion implantation on the structure and optical properties of the nanoscaled surface layer. The impact was placed to the possibility to achieve control over the mechanical stress in the silicon oxide layers since this pa-

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parameter has an effect on overall characteristics of the devices including structural defects.

Hydrogenation of the surface region of c-Si substrates is achieved by shallow low energy plasma-beam H<sup>+</sup> ion immersion implantation with different fluences up to 10<sup>15</sup> cm<sup>-2</sup>. The study was performed using highly sensitive optical methods combined with modeling the distributions of the ions and implantation induced defects. The process-induced changes in the structure and optical properties of the thin hydrogenated Si layer and the SiO<sub>x</sub> layer formed on it are studied.

#### EXPERIMENTAL DETAILS

The wafers in this study were monocrystalline Si (c-Si), Cz-grown Wacker wafers with orientation Si(100) and Si(111) and resistivity ranging from 4 to 10 Ohm.cm. The c-Si surface was modified by plasma immersion ion (PII) implantation accomplished in a planar plasma reactor. No externally heating was applied to the substrates. The energy of H<sup>+</sup> implant was chosen as low as 2 keV, so that the projected H<sup>+</sup> implantation depth in Si substrate to be approximately within 20–30 nm inwards the Si surface. The H<sup>+</sup> fluence varied from 10<sup>13</sup> to 10<sup>15</sup> H<sup>+</sup>/cm<sup>2</sup>. Before implantation the samples underwent a standard RCA cleaning procedure, while after implantation, possible surface contaminations were removed by a short dip in diluted HF.

Next very thin silicon oxide layers have been formed on the hydrogenated Si substrates through exposure to dry O<sub>2</sub> atmosphere at temperatures ranging from 700° to 850°C. The layers were characterized by micro-Raman spectroscopy (RS), spectroscopic ellipsometry (SE) in the VIS range and electroreflectance (ER) spectroscopy. The observed displacement of the characteristic peak at 3.4 eV of Si relative to unstressed situation in the SE and ER spectra is related to internal stress resulting from the technological processes. In the spectra RS the mechanical stress from the shift of the 520 cm<sup>-1</sup> Si Raman phonon mode is known to be sensitive to lattice strain [6]. The Raman spectra were obtained by LabRAM HR micro-Raman spectrometer equipped with CCD detector at laser excitation wavelength of 632 nm. The peak position accuracy is below 0.05 cm<sup>-1</sup>. Using ER and SE the mechanical stress in Si/oxide was determined from the shift of the energy position of the Si direct bandgap at 3.4 eV [7]. The ER spectra were measured recording the modulation of the

reflectivity  $\Delta R/R$  in response of an external modulating electric field applied to the sample in the photon energy region of 3.0–3.8 eV around the Si direct bandgap energy. The sensitivity in measuring  $\Delta R/R$  was 1 ppm, and the accuracy of measuring the signal strength was 2%. The measurements were performed at room temperature in the low-field mode condition. The values of the direct transition  $E_g$  and broadening parameter  $\Gamma$ , were calculated from the analysis of the ER spectra line shape using the Aspnes three-point technique [8]. SE spectra were taken with a “Rudolph Research” variable-angle ellipsometer with polarizer-compensator-sample-analyzer configuration in the spectral range of 300–640 nm. The accuracy of the polarizer, analyzer and incidence angle was within  $\pm 0.01^\circ$ . The pseudo-dielectric function  $\langle \epsilon \rangle$  has been calculated following the procedure outlined elsewhere [9].

The depth profiles of the implanted ions and implantation induced defects were derived using the SRIM code based on a Monte-Carlo simulation method [10] for different ion fluences.

#### RESULTS AND DISCUSSION

Hydrogenation is known for its modification of Si surfaces [11], forming an overlayer which is less dense and contains voids and attached H-atoms and defect sites [12]. On one hand voids could mean smaller internal stress and subsequent oxidation of the modified surface can proceed faster. On the other hand, the transitional region between the oxide and the Si substrate can be located just in this modified layer. Hydrogenation induced defects are precursors of interface defect centers that appear at the interface after dry oxidation. Some defects remain in the Si substrate and some are incorporated into the oxide layer. Although implantation with the light H<sup>+</sup> ions is not expected to cause severe damage in the Si lattice, the ionization effects can be substantial. Simulation using SRIM code shows the hydrogen concentration profile ionization events in the Si subsurface region as displayed in Fig. 1. High hydrogen concentration in the Si subsurface region is evident from the results in Fig. 1a, which however, is not necessarily sufficient for complete hydrogen saturation of the dangling bonds at the interface. The ionization effects lead to defect generation in the Si subsurface region where the oxide layer will be displayed during oxidation. This region is marked in Fig. 1 by the dotted line.

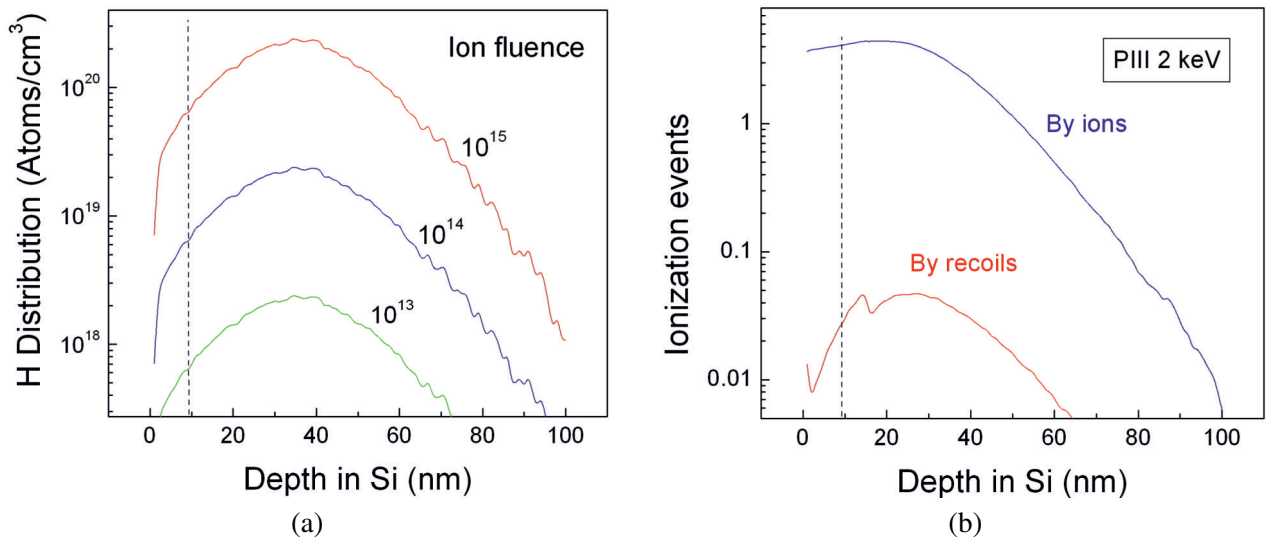


Fig. 1. Ion (a) and vacancy (b) profiles for implantation with 2 keV H<sup>+</sup> into Si wafers.

From the ion distribution in Fig.1a it is evident that the maximum of the concentration is located at 36 nm. During subsequent oxidation when the hydrogenated Si wafer is subjected to elevated temperature this distribution will alter. Suggesting diffusion as a mechanism of migration of the H<sup>+</sup> ions the profile was modeled using standard diffusion equation with  $D = D_0 \exp(-E_a/kT)$  diffusion coefficient  $D_0 = 9.4 \times 10^{-3} \text{ cm}^2/\text{s}$  and activation energy  $E_a = 0.48 \text{ eV}$  [13]. The ion distribution profile was flattened at a very low level. Such a low hydrogen concentration would have no influence on the oxidation process and on the optical and morphological properties of the hydrogenated Si surface. It can be suggested that possible anomalies in H<sup>+</sup> diffusion behavior in Si occur, which can be attributed to trapping of H at implantation induced defect centers. Support of this suggestion comes from modeling of the optical constants from data gained by the ellipsometric study. Performing the optical modeling results have been gained for the thickness of the layer being modified by the hydrogen implant and the alteration of its optical constants from those of unimplanted c-Si material. The optical modeling of the ellipsometric data (discussed in detail elsewhere [14] established a thin native oxide on the Si substrates with an average thickness of 3 nm (Fig. 2a). The thickness of the modified Si layer, given also in Fig. 2a, was found to be dependent on H<sup>+</sup> fluence. It can be seen that it shrinks with increasing the ion fluence. Most probably, higher H<sup>+</sup> fluence caused stronger lattice disorder and larger amount of defects in the Si surface region,

being an impediment to the H motion inwards. The other reason for that could be the H solubility-related saturation of the Si surface, which additionally hinders the H implants motion [15,16]. Indirect evidence for the process induced damage was the presence of an amorphized Si phase in the Si surface layer. Depending on H<sup>+</sup> fluence its volume fraction amounted to 2.4%, 2.7% and 5.8% for fluences of 10<sup>13</sup>, 10<sup>14</sup> and 10<sup>15</sup> cm<sup>-2</sup> H<sup>+</sup>, respectively (Fig. 2b). Including voids fraction in the modeling of the effective medium composition gave unsatisfactory fitting results. This can be regarded as an indication that the voids, if present, bear a lower fraction than the one used in the modeling. This composition can be characterized as substoichiometric SiO<sub>x</sub>.

As next approximation SRIM simulation was performed for the implantation energy of 2 keV used, the implantation being taken to proceed into Si through the native SiO<sub>2</sub> with thickness of 3 nm as found from the SE data modeling. Fig. 3 shows the depth profiles of the implanted H<sup>+</sup> ions and the implantation generated defects (vacancies created by incoming ions and recoils). From Fig. 2 it was inferred that the thickness of the H-modified Si region depends on ion fluence. The question is whether this dependence can be related to the profile of the implanted hydrogens since it is known that different fluences change only the intensity of peaks and not their position.

As seen in Fig. 3a, the deconvolution of the ion concentration spectrum resulted in two components, one extending deeper into the Si bulk. It could be expected that the shallower peak lies in the native oxide.

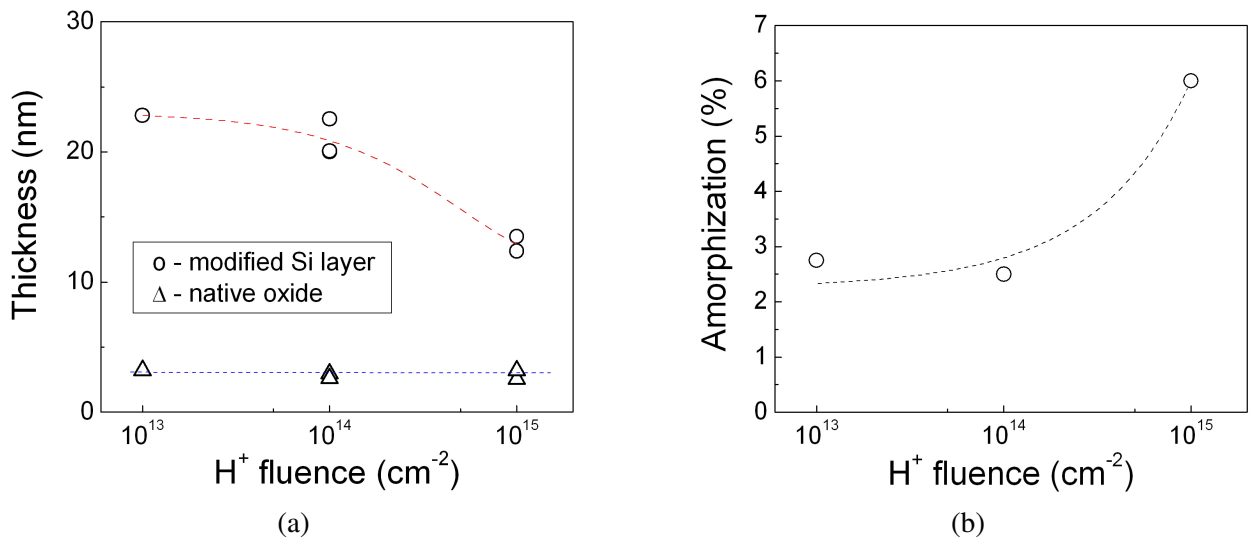


Fig. 2. Thickness of the native oxide and the modified Si surface layer vs.  $H^+$  fluence (a). Volume fraction of the amorphized Si in the modified Si layer (b).

However, this peak has its maximum at a much deeper position ( $\sim 20$  nm) than the  $SiO_2$  thickness ( $\sim 3$  nm). Then it is reasonable to suggest that the modified Si region is related rather to defects created by the ion implantation process than the projected range of hydrogen ions. In Fig. 3b SRIM simulation data for vacancies shows two defect regions that can be identified lying in the oxide and within the Si. The effect of the ion implantation fluence on the modified Si thickness is not obvious. However, it can be suggested that the highest fluence can have a more pronounced effect on the modification process. The implanted hydrogen atoms could be captured in the  $SiO_2$  up to

the peak of the defects in the bulk to form a region with high H-concentration similarly to the process by the Si exfoliation [17]. Taking in view that by PII not only  $H^+$  but also  $H_2^+$  and  $H_3^+$  species are implanted, whose energies are 1/2 and 1/3 of the total implantation energy, respectively. The defect profiles for these species are shallower than for the  $H^+$ , filling the well between the  $SiO_2$  and bulk peaks and forming a continuous defect region. Our fluences are well below the typically needed for film transfer process of mid- $10^{16}$  atoms/ $cm^2$  [11], so that no pile-up takes place, but the highly hydrogenated region can hinder further H-penetration into the Si bulk. The modified Si region

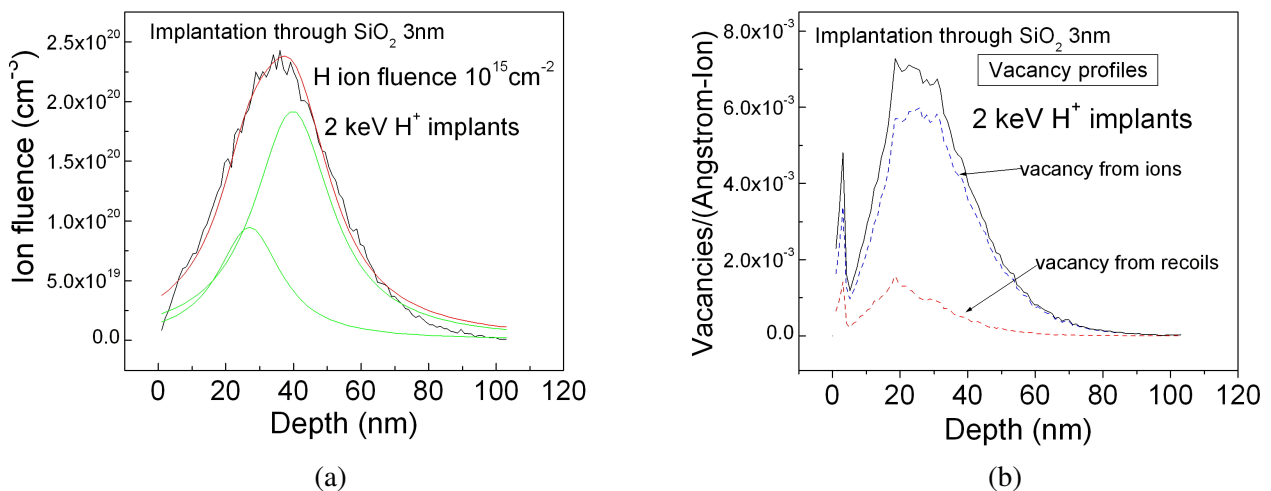


Fig. 3. Ion (a) and defect (b) profiles for implantation with 2 keV  $H^+$  through  $SiO_2$  into Si wafer.

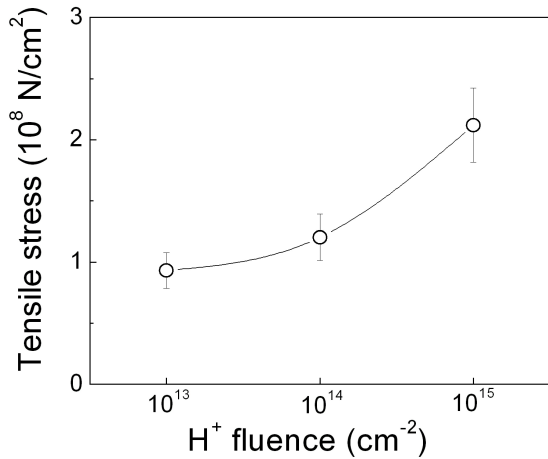


Fig. 4.  $H^+$  implantation induced stress in the native oxide and the Si substrate as a function of  $H^+$  fluence.

would then extent up to about 20 nm as seen by the position of the defect concentration peak in Fig. 3b, which correlates well with the thicknesses of the modified Si surface layer ranging 23-14 nm (Fig. 2a).

Modeling the dielectric function shifts of the critical energy points around  $\sim 3.4$  and  $\sim 4.2$  eV were observed. These were interpreted as due to the structural defects created by the ion implantation process, which in turn generates strains inside the silicon. Therefore, the shifts of the energy positions carry information about the internal stress in the modified layer. The evaluated tensile stress levels in Si as a function of  $H^+$  fluence is presented in Fig. 4. Here the average shifts of the two energy peaks are taken for the stress calculation. Apparently,  $H^+$  implanta-

tion with lower fluences generated negligible structural strains in the implanted region and even at  $10^{15} cm^{-2}$  it remains below  $2.5 \times 10^8 N/cm^2$ .

Modification of Si surface layer upon hydrogenation of the Si wafer influences the subsequent oxidation mechanism. The transitional region between the oxide and the Si substrate can be located just in this modified layer. It was established that oxidation rates increase up to values comparable those for wet oxidation and are orientation dependent. This is evident in Fig. 5, where comparative results on the oxidation rates are displayed for both Si(111) and Si(100).

Mechanical stress levels in the Si surfaces hydrogenated at different conditions were evaluated applying the three spectroscopic techniques SE, RS and ER. The refractive index as a function of wavelength gained from modeling of SE data is presented in Fig. 6a. Taking the values at 633 nm the stress was calculated. From the shifts of Si Raman mode (Fig. 6b) and Si direct energy gap in ER spectra (Fig. 6c) relative to unstressed positions also allow information on stress level to be extracted. The quantification to transfer the SE, RS and ER results into mechanical stress was assessed taking in view the strain coefficient corresponding to theoretically evaluated hydrostatic equivalence [18,19], assuming homogeneous structures and, consequently, uniaxial stress conditions. Evaluating the stress from ER and SE correlation coefficient was taken  $2.09 \times 10^7 N/m^2/meV$  [20]. Applying RS the value of  $4.34 \times 10^8 N/m^2/cm^{-1}$  was used [21]. The results are summarized in Fig. 6d.

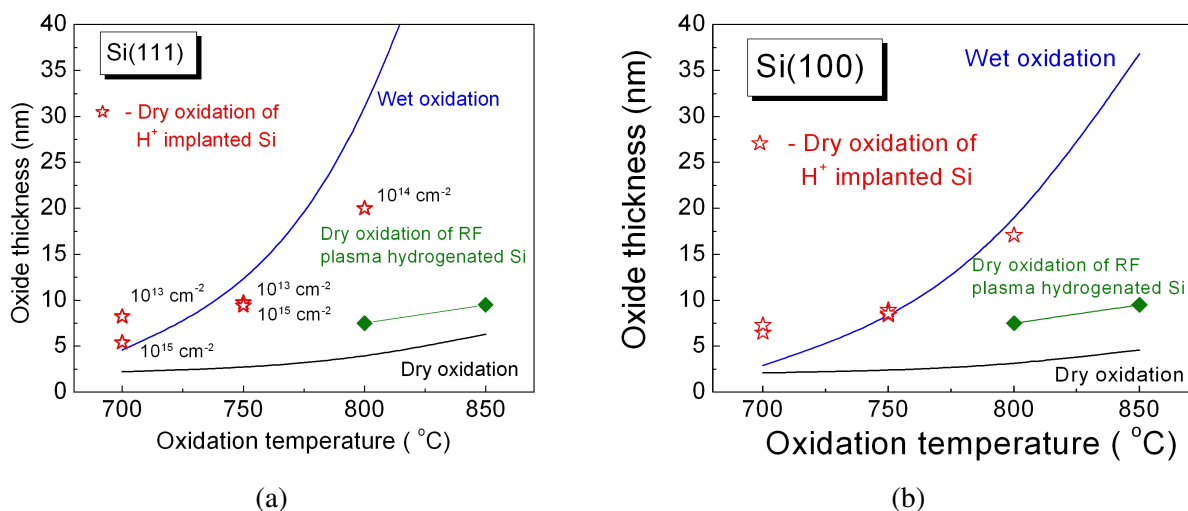


Fig. 5. Oxidation rates for Si wafers hydrogenated with different  $H^+$  fluences at oxidation temperatures from 700 to 850 $^{\circ}C$  for Si(111) (a) and Si(100) (b).

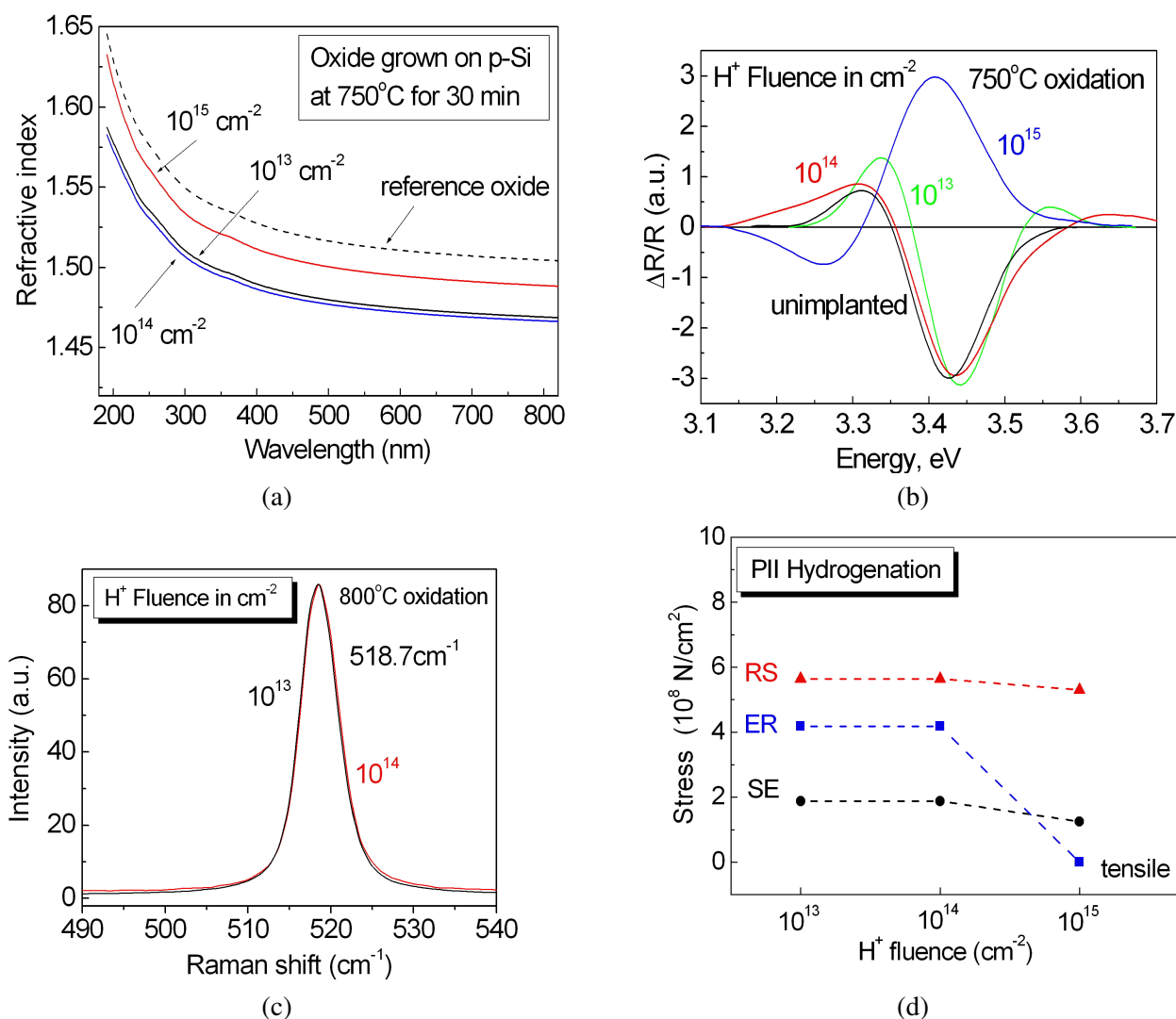


Fig. 6. a) SE spectra of the refractive index of the oxidized Si surface; b) Electroreflectance spectra of the oxidized Si surface for different  $H^+$  implantation fluences; c) RS spectra for oxidized Si surface hydrogenated by RF hydrogen plasma; d) Mechanical stress in oxidized Si surface hydrogenated by PIII.

Additional conclusion from the results for the refractive index in Fig. 6a concerns the value at 633 nm, which is below the typical 1.46 for stoichiometric  $\text{SiO}_2$ . This supports the assumption that oxide composition is  $\text{SiO}_x$ ,  $x < 2$ .

Although certain differences are observed in the results gained by the different measurement techniques, there are tendencies that are worth to notice. It is well documented that thermal oxidation of Si results in oxide in compressive stress and, consequently tensile in the underlying Si. The optical techniques applied in the present study probe the stress condition of the Si substrate. It is seen from Fig. 6d that hydrogenation results in low tensile stress of Si and low compressive stress in the oxide, i.e. leads to less

strained oxide/Si structure in comparison to over  $10^9 \text{ N/cm}^2$  for oxide on unhydrogenated Si [22]. Lower stress level is related to low defect densities.

Comparison with the stress level after PII hydrogenation (see Fig. 4) shows that oxidation process induces additional stress, which is due to the extension of the Si lattice parameter through the advent of the oxygen atoms. It is well established that this enlargement amounts to about 20% from the initial Si volume. In spite of the oxidation induced increase, the obtained stress levels show low levels taking in view that the oxidation temperature is low and no postoxidation annealing was applied. This beneficial effect is achieved through Si hydrogenation by PIII technology.

## CONCLUSIONS

Application of PII implantation on Si wafers modifies the Si surface creating a 14-23 nm thick (varying with H<sup>+</sup> fluence) Si surface layer characterized by low degree of amorphization (up to 5.8 %), creation of structural defects and small degree of internal tensile stress. The modified Si region is related rather to defects created by the ion implantation process than the projected range of hydrogen ions. The subsequent oxidation at low temperatures below 800°C results in growing of substoichiometric SiO<sub>x</sub> layer with low compressive stress. Oxidation rate is higher in comparison to dry Si oxidation approaching the value for wet oxidation. Modification of Si by PIII hydrogenation offers possibility of preparation of SiO<sub>x</sub> layers on Si wafers at controllable mechanical stress at technologically reasonable oxidation rate.

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МОДИФИЦИРАНЕ НА Si ПОВЪРХНОСТ ЧРЕЗ ПЛАЗМЕНА ЙОННА ИМПЛАНТАЦИЯ  
НА  $H^+$  И ТЕРМИЧНО ОКИСЛЕНИЕ

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(Резюме)

Модификацията на полупроводникови повърхности представлява интерес за приборите от следваща генерация, при които активните области се разполагат в тънък приповърхностен слой. Включването на водород в силиций дава възможност за формиране на свръхтънки наноразмерни слоеве от силициев оксид, които могат да изпълняват различна роля в структури и прибори на базата на силиций.

В настоящата работа са представени резултати по изследване на оптичните свойства на окислена Si повърхност, модифицирана чрез хидрогениране на тънък приповърхностен слой. Хидрогенирането се извършва чрез плазмена йонна имплантация на  $H^+$  йони с енергия 4 keV и дози от  $10^{13} H^+/cm^2$  до  $10^{15} H^+/cm^2$ . Свръхтънки слоеве  $SiO_x$  се формират върху хидрогенираната Si повърхност чрез окисление в среда на сух  $O_2$  при температури от 700°C до 850°C. Словете са характеризирани чрез микро-Раманова спектроскопия, спектрална елипсометрия във видимата област и електроотражение. Изследвани са пряката зона при 3.4 eV на Si от данните на спектрална елипсометрия и електроотражение, параметърът на уширение на пиковете на електроотражение и положенията на Рамановите линии и полуширините им. Отместванията на характеристичните пикове в изследваните спектри са пряко свързани с вътрешните механични напрежения в структурите. Резултатите показват, че върху хидрогенирана Si повърхност могат да се получават оксидни слоеве с понижени механични напрежения и високи концентрации на структурни дефекти.

При плазмената имплантация модифицираният слой е с дебелина от 14 до 23 nm в зависимост от  $H^+$  доза и се характеризира с ниска степен на аморфизация (до 5.8%), генерация на структурни дефекти и ниски механични напрежения на разтягане. Параметрите на оксидните слоеве също показват зависимост от имплантационната доза, а също и от температурата на окисление. Вариациите в състава и оптичните параметри на слоевете са свързани със структурата на модифицирания при имплантацията Si слой. Процесът на окисление води до ниски механични напрежения на свиване (под  $\sim 7 \times 10^8 N/m^2$ ) и показател на пречупване, по-нисък от този на стехиометричния  $SiO_2$ . Специално внимание е отделено на възможностите за контрол на механичните напрежения в слоевете, тъй като те влияят на структурните дефекти и по този начин определят крайните характеристики на приборите.