

# Surface morphology of RF plasma immersion H<sup>+</sup> ion implanted and oxidized Si(100) surface

M Anastasescu<sup>1</sup>, M Stoica<sup>1</sup>, M Gartner<sup>1</sup>, S Bakalova<sup>2</sup>, A Szekeres<sup>2,4</sup>  
and S Alexandrova<sup>3</sup>

<sup>1</sup>Institute of Physical Chemistry, Romanian Academy of Sciences,  
202 Spl. Independentei, 060021 Bucharest, Romania

<sup>2</sup>Acad. G. Nadjakov Institute of Solid State Physics,  
Bulgarian Academy of Sciences, 72 Tsarigradsko Chaussee, 1784 Sofia, Bulgaria

<sup>3</sup>Technical University of Sofia, 8 Kl. Ohridski Blvd., 1797 Sofia, Bulgaria

E-mail: szekeres@issp.bas.bg

**Abstract.** The surface morphology of p-Si(100) wafers after RF plasma immersion (PII) H<sup>+</sup> ion implantation into a shallow Si surface layer and after subsequent thermal oxidation was studied by atomic-force microscopic (AFM) imaging. After PII implantation of hydrogen ions with an energy of 2 keV and fluences ranging from 10<sup>13</sup> cm<sup>-2</sup> to 10<sup>15</sup> cm<sup>-2</sup>, the Si wafers were oxidized in dry O<sub>2</sub> at temperatures ranging from 700 °C to 800 °C. From the analysis of the AFM images, the surface amplitude parameters were evaluated and considered in terms of the technological conditions. The amplitude parameters showed a clear dependence on the H<sup>+</sup> dose and the oxidation temperature, with the tendency of increasing with the increase of both the H<sup>+</sup> ion fluence and the oxidation temperature. The implantation causes surface roughening, changing the RMS roughness value from 0.15 nm (typical for a polished Si(100) surface) to the highest value 0.6 nm for the H<sup>+</sup> fluence of 10<sup>15</sup> ions/cm<sup>2</sup>. Oxidation of the H<sup>+</sup> implanted Si region, as the oxide is growing inward into Si, levels away the pits created by implants and results in a smoother surface, although keeping the RMS values larger than 0.2 nm.

## 1. Introduction

The growth of reliable thin thermal oxide on silicon in a thin-film regime is important for fabrication of smaller and faster MOS device structures. Growth of low-defect ultra-thin oxides with improved electrical and structural properties can be achieved at suitable oxidation rates and lowered oxidation temperatures by hydrogenation of silicon prior to the oxidation process. In the Si technology, hydrogenation is usually achieved by an implantation process, either beam [1,2] or plasma ion implantation (PIII) [3]. During the implantation process, due to plasma radiation and collisions of implanted ions with the surface and atoms of the substrate material, lattice defects are created, which is a shortcoming in view of preparing advanced MOS structures for the future nanoelectronics. On the other hand, ultrathin SiO<sub>2</sub> with easily controllable thickness, high dielectric quality and perfect interfaces is difficult to be obtained at temperatures lower than

<sup>4</sup> To whom any correspondence should be addressed.



900 °C. Moreover, the pre-oxidation conditions of the Si surface play a decisive role and, hence, will influence the growth kinetics of ultrathin oxides and their properties. All of the above calls for comprehensive studies of the surface, oxide and interface properties in relation with the technology of MOS device preparation.

Our recent research has been focused on the growth of high-quality ultra-thin SiO<sub>2</sub> layers with decreased mechanical stress at rather low oxidation temperatures through hydrogenation of the near surface region of Si wafers prior to oxidation. We have established that thin SiO<sub>2</sub> layers can be grown on Si and hydrogenated by either RF plasma immersion ion implantation or RF plasma treatment, thus reducing the structural strain and the concentration of defects [4,5].

In the present paper, results from the study of the surface morphology of p-Si(100) wafers created by RF plasma immersion implantation (PII) of H<sup>+</sup> ions into a shallow silicon surface layer followed by its thermal oxidation at temperatures below 850 °C. From the analysis of the atomic-force microscopy (AFM) results, data on the surface amplitude parameters were obtained and considered in terms of the technological conditions. The AFM observations suggest the creation of a thin, less dense, Si surface region due to the formation of voids by the hydrogen particles incorporated into the growing oxide layer. The AFM results show that, during oxidation, a surface flattening takes place probably due to faster Si oxidation facilitated by the H<sup>+</sup> implants along hillocks and pits leading to a more homogeneous surface morphology.

## 2. Experimental details

The samples studied were Cz-grown p-type Si Wacker wafers with orientation (100) and a resistivity of 5 – 8 ohm cm. All wafers were subjected to a standard RCA cleaning procedure before any technological steps. Part of the samples were loaded into a planar plasma reactor and were exposed to plasma-beam ion implantation of hydrogen with an ion energy of 2 keV and fluences varying from 10<sup>13</sup> to 10<sup>15</sup> cm<sup>-2</sup>. No external heating was applied to the Si substrates. The low energy of the H<sup>+</sup> ions (2 keV) allowed to implant them in a shallow depth in the Si substrate, namely, approximately within 20 – 30 nm inwards the Si surface. After implantation, possible surface contaminations and native oxide were removed by a short dip of the samples in diluted HF.

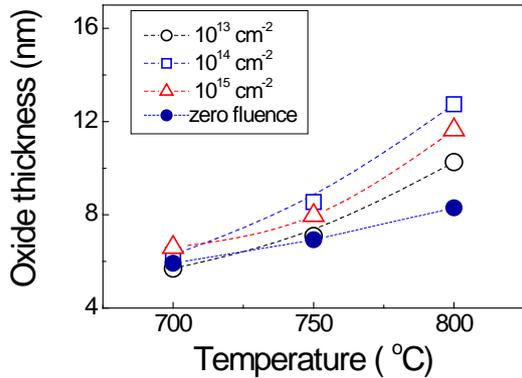
Further, the hydrogen implanted Si wafers were oxidized in a dry O<sub>2</sub> atmosphere for 30 minutes at temperatures of 700 °C and 750 °C and for 20 minutes at 800 °C. In the same oxidation runs, unimplanted Si wafers were simultaneously oxidized to serve as a reference. Choosing these oxidation durations and taking into account the oxide growth kinetics at standard dry oxidation, we expected that the oxide thicknesses will be in the same range.

We should emphasize that no further thermal treatment was applied to the studied samples.

The topography measurements were performed before and after oxidation of the Si wafers under ambient conditions on a XE-100 apparatus from Park Systems (2011) in non-constant force mode. The decoupled XY-Z scanners with out-of-plane motion of less than 1 nm over a 50-micron scanned area allowed measurement of the surface roughness with an accuracy of less than 1 nm. Ultra-sharp tips (< 8 nm tip radius; PPP-NCLR type from Nanosensors<sup>TM</sup>) working with a spring constant of 48 N/m and a resonance frequency of ~190 kHz were used in the measurements. The sample surfaces were scanned over an area of 1×1 μm<sup>2</sup> and the XEI program was used for the statistical data analysis. From the 2D and 3D AFM imaging, the amplitude parameters, namely the peak-to-peak height,  $S_p$ , the root-mean-square (RMS) roughness,  $S_q$ , the surface skewness,  $S_{sk}$  and the surface kurtosis,  $S_{ku}$ , were determined.

## 3. Results and discussion

The thickness of the grown SiO<sub>2</sub> layers, determined by ellipsometric measurements with an accuracy of ± 0.2 nm, varied within 5 – 14 nm depending on the H<sup>+</sup> implant fluence and the oxidation temperature. This is illustrated in figure 1, where the SiO<sub>2</sub> thicknesses are summarized. In comparison to standard SiO<sub>2</sub> grown on unimplanted Si, thicker oxides grew into H<sup>+</sup> implanted Si as the thickness difference increased with raising the oxidation temperature. Obviously, introduction



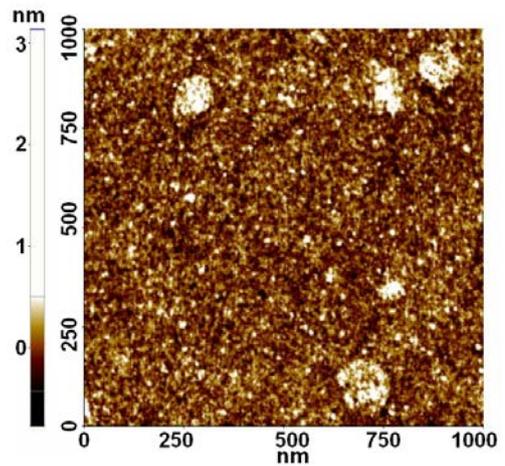
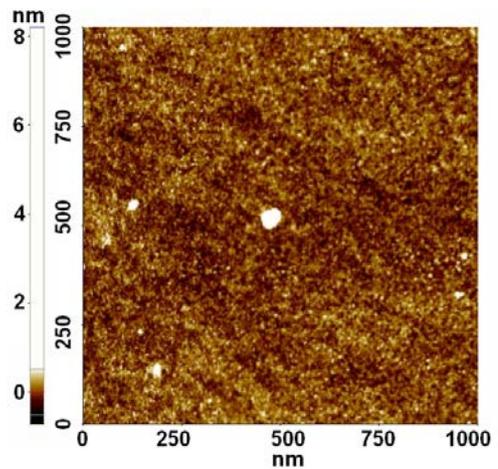
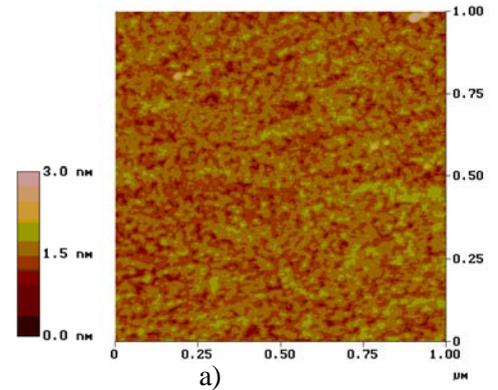
**Figure 1.** SiO<sub>2</sub> thickness versus oxidation time for (100) Si implanted with H<sup>+</sup> ions with different fluences (inset). For comparison purposes, the thickness of SiO<sub>2</sub> layers grown into unimplanted (100) Si wafers is also given. In the ellipsometric measurements, the thickness values are obtained with an accuracy of ± 0.2 nm.

of H<sup>+</sup> and the plasma radiation create favourable conditions for accelerated oxide growth kinetics.

The surface topographic study of bare Si wafer revealed a highly smooth good-quality surface with a RMS roughness of 0.15 nm typical for polished commercial Wacker wafers. It is well seen in figure 2a that the initial silicon surface is flat with few randomly distributed hillocks. Because the AFM measurement was made in air, the appearance of hillocks on the flat surface could be connected with native SiO<sub>2</sub> with a thickness of ~2 nm re-grown on Si top.

Low-energy shallow implantation of hydrogen ions into the wafers does not worsen essentially the Si surface flatness, but creates bending and a large number of deep pits. The influence of implantation becomes stronger when the H<sup>+</sup> fluence is higher. The overall effect is that Si surfaces subjected to PII implantation have larger amplitude parameters values with a trend to increasing as the ion fluence is increased. The AFM observations suggest the creation of a thin, less dense, Si surface region due to the formation of voids by the hydrogen particles incorporated into the near-surface Si region. The existence of a less dense region facilitates the diffusion of oxidants and, thus, a higher Si oxidation rate is only to be expected, as was observed (figure 1).

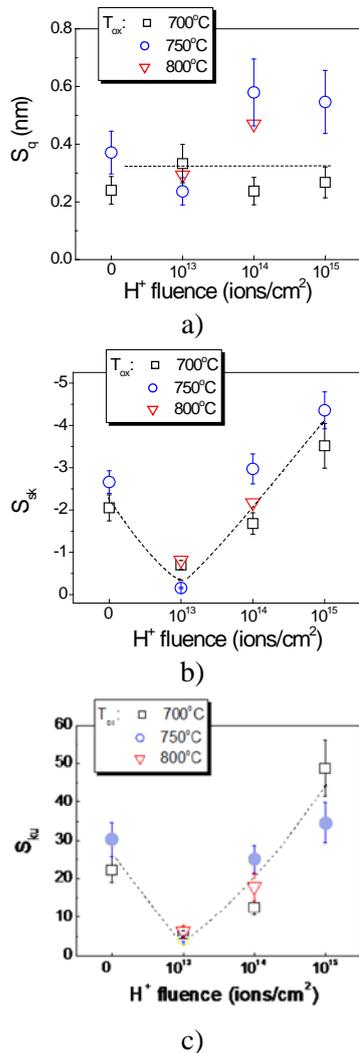
A typical AFM image taken from an implanted Si surface is presented in figure 2b for the H<sup>+</sup> fluence of 10<sup>14</sup> ions/cm<sup>2</sup>. A large number of pits are visualized on the considerably flat surface.



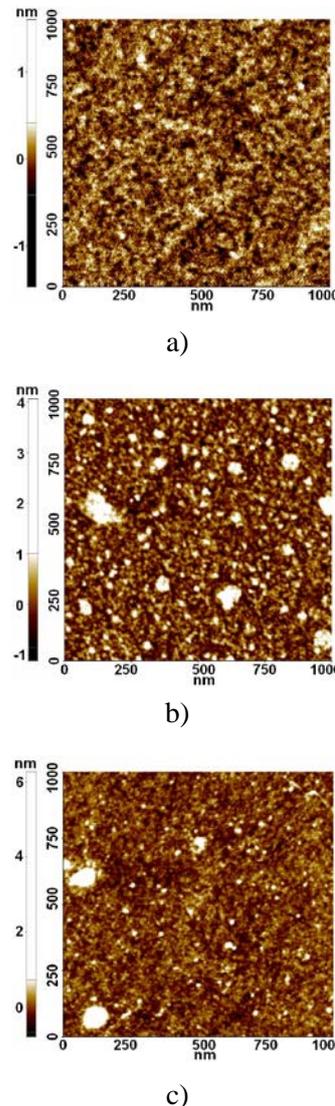
**Figure 2.** AFM 2D surface images of (100)Si substrate before (a) and after PII implantation of hydrogen with a fluence of 10<sup>14</sup> ions/cm<sup>2</sup> (b) followed by thermal oxidation at 700 °C (c).

This is reflected in the slight increase of the RMS value to 0.265 nm, in the large negative skewness ( $S_{sk} = -14.139$ ) and the extremely high positive kurtosis ( $S_{ku} = 350.591$ ) values. The phase contrast made from the same AFM picture (not shown) pointed out that the big white spot in the middle of the image in figure 2b is dust. We would like to note that this image of dust on the surface is intentionally given for comparison purposes, because similar features appeared in many of AFM images but, as it turned out later, they could not be related to dust, as will be discussed below. The effect of oxidation on the surface morphology is illustrated in figure 2c and will also be discussed below.

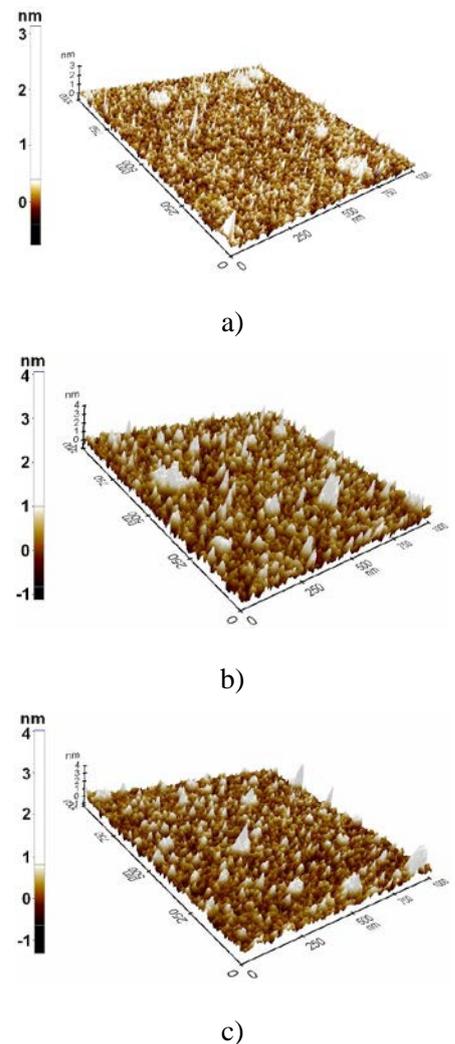
The AFM amplitude parameters, calculated from the scanned area of the oxidized Si surfaces, showed a clear dependence on the  $H^+$  fluence and the oxidation temperature. Their values are



**Figure 3.** Dependence of the amplitude parameters, namely, root-mean-square (RMS) roughness,  $S_q$  (a), surface skewness,  $S_{sk}$  (b) and surface kurtosis,  $S_{ku}$  (c) of oxidized Si surface on the  $H^+$  ion fluence; the oxidation temperatures are given in the inset.



**Figure 4.** AFM 2D surface images of  $SiO_2$  layers grown at 750°C on (100)Si, implanted at different  $H^+$  fluences:  $10^{13}$  ions/cm<sup>2</sup> (a);  $10^{14}$  ions/cm<sup>2</sup> (b) and  $10^{15}$  ions/cm<sup>2</sup> (c).



**Figure 5.** AFM 3D surface images from  $1 \times 1 \mu m^2$  scanned surface area of  $SiO_2$  layers into (100)Si, implanted with  $H^+$  fluence of  $10^{14}$  ions/cm<sup>2</sup>. The oxides were grown at temperature of 700°C (a), 750°C (b), and 800°C (c). The  $\Delta z$  scale is 3 nm (a) and 4 nm for (b) and (c).

summarized in figure 3. The surface of the SiO<sub>2</sub> grown on unimplanted Si wafers is considerably smooth. The large kurtosis, however, indicates a “spikiness” of the selected oxide surface.

Oxidation of the H<sup>+</sup> implanted Si region, as the oxide is growing inwards the Si, levels away the pits created by implants and results in a more homogeneous and smoother surface, albeit keeping the RMS values larger than those of the initial Si surface. The temperature treatment (oxidation) itself releases the structural strain caused by the implantation process, as it is suggested from the significant lowering of the 3D surface parameters ( $S_{sk}$  and  $S_{ku}$ ) values. However,  $S_{sk}$  and  $S_{ku}$  still have considerably large values indicating a strong peakedness of the surface height distribution and the predominance of a valley structure comprising the surface.

For comparison purposes, figure 2c presents the 2D AFM image of a Si surface region PII implanted with a H<sup>+</sup> fluence of 10<sup>14</sup> ions/cm<sup>2</sup> and oxidized at 700 °C. Randomly distributed hillocks and pits are observed on the rather flat surface (RMS = 0.26 nm). The phase contrast picture of the same AFM image revealed that the large-size spherical-like features could not be related to dust, as was the case in figure 2b, but rather to the material. They arise from the deformations of the implanted surface by thermal treatment (oxidation) and are H-induced surface blisters. Hydrogen implantation-induced surface blistering/exfoliation has been observed earlier [6] and has been further developed as an ion-cut process in direct wafer bonding techniques [7,8]. In our case, the effect of surface blistering is enhanced by increasing both the H<sup>+</sup> ion fluence (figure 4) and the oxidation temperature (figure 5). Although the accelerated oxidation process at elevated temperatures (figure 1) smoothens out the surface, the above-mentioned effect could lead to the minimum in the surface parameters values observed in figure 3.

As the H<sup>+</sup> fluence is increased, the surface roughness of the SiO<sub>2</sub> layer slightly increases (figures 3 and 4). The close peak-to peak ( $S_y$ ) values and the more negative skewness ( $S_{sk}$ ) indicate that the features on the surface are more randomly distributed. When the oxidation temperature is raised, the number of randomly distributed and sized hillocks and pits shows a tendency to increase (figure 5), leading to an increase of the surface roughness (a larger  $S_q$ ) and a growing number of pits (a more negative  $S_{sk}$ ).

The AFM results show that the temperature treatment (oxidation) releases the structural strains caused by the implantation process, as it is suggested from the significant lowering of the 3D surface parameters values. However, the  $S_{sk}$  and  $S_{ku}$  parameters still have considerably large values indicating a strong peakedness of the surface height distribution and the predominance of a valley structure comprising the surface. The negative values of the surface skewness are indicative for the existence of superficial voids, which may be caused by both implantation (ion bombardment) and surface reorganization (relaxation and re-ordering during thermal treatment).

#### 4. Conclusions

The surface quality of the optimized ultra-thin oxide grown by preceding hydrogenation of Si wafers is an important milestone for further device fabrication. From the analysis of the AFM images of the samples surface, the following conclusions are drawn: (i) the surface of the RF PII implanted Si wafers have a relatively flat top and valleys; (ii) the subsequent thermal oxidation of the implanted Si wafers at temperatures of 700 °C, 750 °C and 800 °C reduces the number and size of pits on the surface but increases the average surface roughness as the temperature is raised and the valley structure comprising the surface still remains predominant. It is suggested that the surface morphology observed of H<sup>+</sup> implanted Si facilitates the diffusion of oxygen atoms into Si and, hence, leads to a larger oxidation rate and a lower level of oxide stress even at temperatures as low as 700 °C.

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