Evolution of the interface structure of bonded Si wafers after high temperature annealing

Journal of Physics: Conference Series **209** (2010) 012058 doi:10.1088/1742-6596/209/1/012058

Evolution of the interface structure of bonded Si wafers after high temperature annealing

ND Zakharov[1], E Pippel[1], P Werner[1], U Gösele[1], V Vdovin[2,3], M Milvidskii[3], M Ries[4], M Seacrist[4] and R Falster[5]

[1] Max Planck Institute of Microstructure Physics, 06120 Halle (Saale), Germany
[2] Institute for Chemical Problems of Microelectronics, 119017 Moscow, Russia
[3] Institute of Rare Metals 'Giredmet', 119017 Moscow, Russia
[4] MEMC Inc, 501 Pearl Drive, St Peters, MO, 63376, USA
[5] MEMC Electronic Materials SpA, Viale Gherzi 31, I-28100 Novara, Italy

ABSTRACT: The evolution of the interfaces of bonded Si wafers and the corresponding low-angle twist boundary have been analysed in dependence on thermal annealing. Two orientation relations were investigated: i) Si(001)/SiO_2/Si(001) and ii) Si(110)/SiO_2/Si(001). The interfaces were analysed by TEM and STEM/EDXEELS. It is found that the decomposition rate of the intermediate oxide layer and the formation of a Si-Si bonded interface depend very much on the lattice mismatch and on the twist angle. A dissolution of the oxide and the formation of Si-Si boundaries occur much faster in the case of Si(110)/Si(001) bonding than in Si(001)/Si(001). The process of interface fusion and the dissolution of the oxide layer are discussed.

1. Introduction

Tensile strain in silicon channels in complementary metal-oxide-semiconductor (CMOS) devices results in enhanced mobility of both electrons and holes [1]. Direct Wafer Bonding (WB) is a promising way to realize this effect in new types of electronic devices [2]. The formation of a perfect crystalline transition at the interface between the wafers is a crucial problem in this technique [3-5]. Thin SiO_2 layers (native oxide of the wafer surfaces) might hamper this fusion process [6]. In the present work the atomic structure and the composition of Si(110)/SiO_2/Si(001) and Si(001)/SiO_2/Si(001) wafer bonded interfaces were studied by transmission electron microscopy (TEM) as well as by scanning transmission electron microscopy (STEM). The behaviour of intermediate native oxide layers during a high temperature annealing and the interfacial atomic structure are the main issues of this work.

2. Experimental

Samples were fabricated by the direct hydrophilic WB of Si wafers still containing a thin native oxide on their surfaces. The as-bonded structure represented in Fig.1 had the following configuration: i) Si substrates of specific orientations, ii) 2 nm thick SiO_2 layer, and iii) 140-nm thick Si layer. Such samples were thermally annealed in the temperature range 1150 to 1200 °C in vacuum. We could observe a decomposition of the SiO_2 interface layer due to out-diffusion of oxygen and the formation of specific Si(110)/Si(001) and Si(100)/Si(001) interfaces. The bonded structures were investigated by TEM, STEM annular dark field (ADF) using a JEOL JEM4010 and an FEI Titan80-300 electron microscope operating at 400 and 300 kV, respectively. The later one was equipped with an imaging C_s corrector, a monochromator and a Gatan energy loss spectrometer allowing the analysis of the composition with spatial and energy resolutions 0.1 nm and 150 meV, respectively.

3. Results and Discussion

3.1 Si(110)/Si(001) bonding

Fig. 2 shows a typical example of bonded Si wafers after thermal annealing. In this particular case the sample was heated just after bonding at 1150 °C for 15 min. The interface is characterised by a 2 nm thin oxide SiO_x layer between the Si substrate and 140 nm thick Si layer. Beside the thin oxide layer, we found regions where the crystal lattices of both wafers had direct contact (see Fig. 3a).

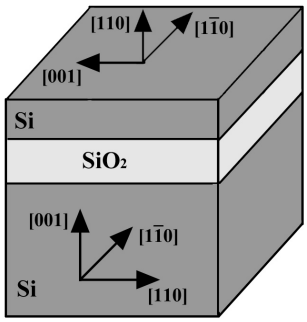

Fig. 1. Schematic view of the bonded structure. Thicknesses of the upper Si layer and the SiO_2 layer amount to 140 nm and 2 nm, respectively.

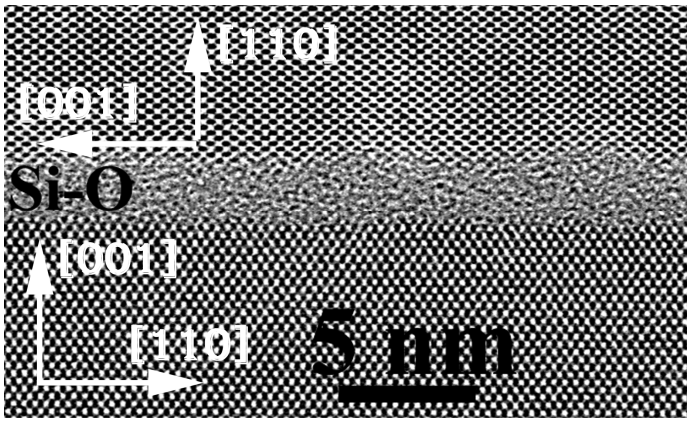

Fig. 2. Cross-sectional image of a Si(110)/Si(001) bonded structure after 15 min annealing at 1150 °C.

This indicates that the oxide layer was partly dissolved ($SiO_x \rightarrow Si + ½ \times O_2$), where the dissociated oxygen could partly diffuse through the 140 nm Si layer and/or to other regions of the interface. In the case of an annealing at 1200 °C for one hour we observed i) extended regions of Si (110)/Si (100), with a directly bonded interface and ii) a rather high number of SiO_x inclusions (see Fig. 3b). From plan-view images it can be shown that these inclusions have a rod-like shape elongated along <110>

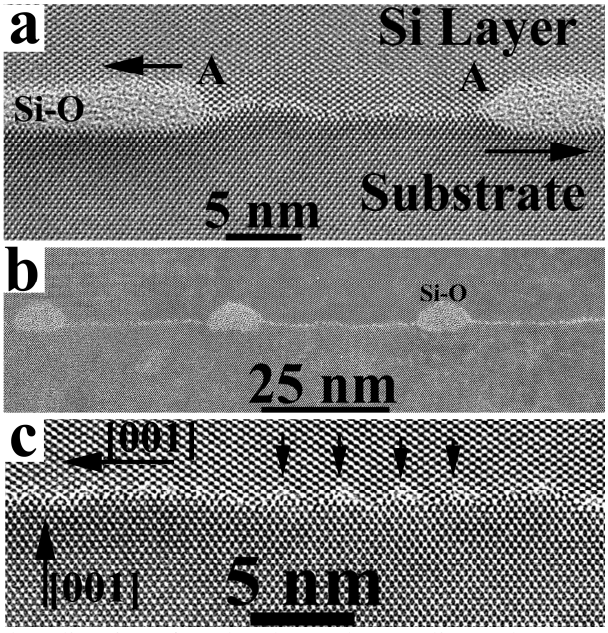

Fig. 3. Evaluation of the bonding interface by thermal annealing. The cross-section images show the Si(110) ‖ Si(001) interface after 15 min (a), 1 hour (b) and 2 hours (c) annealing at 1200°C. The SiO_x inclusions decrease (a+b) and triangular lattice features are generated (arrows in c).

directions in this particular case. The reason for this behaviour might be that in this direction both crystal lattices have the best fit. Annealing of the bonded structure during 2 hours at 1200°C results in the formation of a Si(110)/Si(001) interface almost free of inclusions (see Fig. 3c).

Analysis by electron energy-loss spectroscopy (EELS) performed with sub-nm spatial resolution reveals a high oxygen concentration in the rod-like inclusions. However, in the directly bonded interfaces (see Fig. 4) no oxygen could be detected within the resolution limit of the EELS measurement (≈ 1 %). Figures 4a and b represent STEM images taken from a relatively thick specimen region (> 15 nm) under high-angle annular dark field (HAADF, a) and low-angle annular dark field (LAADF, b) conditions, respectively. The occurrence of a bright line area in the LAADF image (b) reveals the existence of highly strained regions. According to [7] this contrast feature is caused by a random strain field, i.e. a random shift of the atoms away from the equilibrium position within the related column in beam direction. The resulting de-channelling causes a scattering of the electron waves in small angles only. Thereby bright image details are generated in the LAADF image. In the present case the thickness of the distorted area at the interface amounts to about 1 nm.

Two further findings should be mentioned. First, from the crystallography of the present Si(001)/SiO_x/Si(001) interface follows a huge lattice mismatch parameter of the atoms in the respective surface planes of 34%, which theoretically would demand misfit dislocations with a distance of about 1 nm. However, in the STEM high-resolution micrographs of the crystal lattice structure annealed for 2 hrs we observed triangular structures with a periodicity of about 2.5 nm as marked by arrows in Fig. 3c. The analysis revealed rods along <110> with side facets of low index {111} planes. These structures probably enable a reduction of the interface energy. In this particular case the bonding configuration is characterized by an arrangement of the dislocation cores in the side facets.

Second, we observed a dissolution of the thin SiO_x layer during annealing and the formation of a direct contact between the two Si lattices. This is shown in Fig. 3a, where the direct lattice contact area A-A was increasing. For longer annealing times the number and sizes of SiO_x precipitates decreased (Fig. 3b) and, at the end, vanished (Fig. 3c). Concerning this process we assume that the SiO_x is decomposed as mentioned above: oxygen is diffusing out through the 140 nm thick cap layer and self-interstitials diffuse from the surface to the interface filling up the volume of the precipitates.

3.2 Low twist angle Si{001)/Si(001) bonding

Contrary to the previous example, where the oxide layer disappeared after a nearly one hour annealing at 1200 °C, in the case of low-twisted boundaries the oxide layer does not remarkably change. A plan-view image of a bonded sample after 1.5 hrs annealing at 1200 °C is shown in Fig. 5. Moiré fringes are formed due to the double diffraction of electrons on {220} atomic planes. The outcome of this is a twist angle of $\varphi = 0.18°$, which serves in our case as a misfit parameter. This value is very small; all previous investigations were performed at much larger twist angles [2, 3]. Surprisingly, the oxide layer was retained and its thickness was reduced only by about 0.2 nm. This might correlate to a reduction of SiO_2 decomposition rate.

At the same time a large number of small round and square shaped regions appear – a direct bonding occurs (see A in Fig. 5a). Inside these regions the crystal lattice of the Si cap layer is rotated at an angle $\gamma = -0.18°$ to fit the substrate (compensation of φ). The maximal radius r of these coherently coupled regions never exceeded 120 nm. This gives us the maximal value of tangential elastic displacement on contour of this area $\tau = r \tan\varphi = 0.38$ nm. This value does not exceed the Burgers vector of a screw dislocation, $b = 0.38$ nm. This fact explains the absence of dislocations inside such areas which can be nucleated only if $\tau > b$. The formation of such coherently bonded regions results in the appearance of three-dimensional elastic strain fields in the multilayer system. They are associated with an additional elastic energy proportional to the square of the misfit parameter ~ $n\varphi^2$, where n relates to the number of such features per square area.

4. Conclusions

The dissolution of thin oxide layers at the interface of hydrophilicly bonded Si wafers strongly depends on their orientation or their azimuth: it is faster in the case of Si(110)/Si(001) bonding than in the case of Si(001)/Si(001). The dissolution of SiO_2 can proceed in two ways: (i) perpendicular to the interface – normal growth, and (ii) in the interface plane – tangential dissolution. In the case of Si(110)/Si(001) bonding the process (ii) seems to be much faster than the normal one. The Si layer is growing from SiO_2 supersaturated by Si atoms. The supersaturation is supplied by the decomposition $SiO_2 \Rightarrow Si + 2O$ at 1200 °C. The growing surface is clearly faceted by close packed {111} planes (see

A in Fig. 3a). The extremely high density of misfit dislocation is due to the huge lattice mismatch of ε= 34%. This can also stimulate a fast growth due to dislocations interacting with point defects.

In the case of low-angle twist bonding of Si(001)/Si(001) the dissolution of SiO_2 proceeds very slowly and the formation of the crystalline bonded interface corresponds to the traditional process. The small regions of coherent bonding have a diameter which is always below the critical one. These regions cannot overcome the energy barrier to nucleate screw dislocation.

In summary, oxygen layers of bonded interfaces can disappear much faster when the misfit between both crystal lattices is large. The decomposition rate of oxide layers is slowed down in cases where the interface region contains a high lattice distortion (stored elastic energy). In the present work one has to consider that the second wafer always was quite thin (thickness of 140 nm) in all experiments allowing an interaction/diffusion with the adjacent specimen surface.

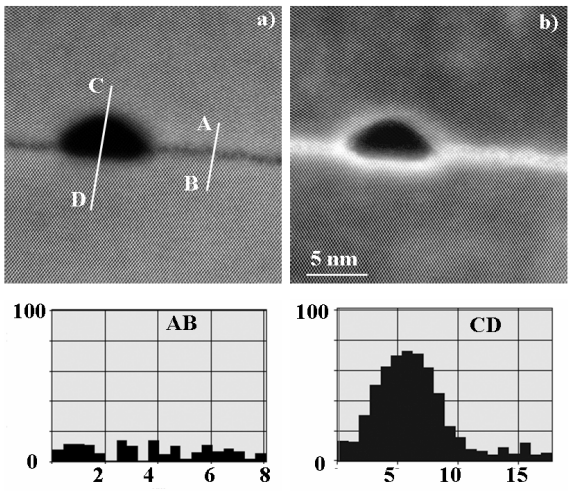

Fig. 4. Images (a) (b) were taken in STEM mode under HAADF and LAADF imaging conditions, respectively. The bright line in (b) indicates the distorted area.
The curves in the lower part represent EELS-profiles of the oxygen concentration (in %) relative to silicon along the marked lines (length in nm).

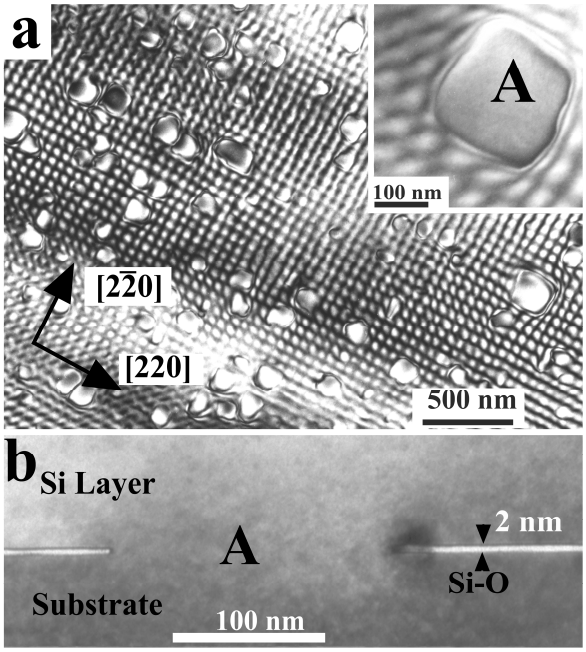

Fig. 5. (a) Plan-view image of Si(001)/Si(001) bonded sample after 2 hrs annealing. Moiré fringes form a square lattice. The regions of coherent coupling of crystal lattices are marked by A. An enlarged image of a coherent region is shown in the upper right corner. (b) Cross-sectional image of coherent regions 'A'. There are no structural defects at the place of bonding.

References

[1] Sato T, Takeishi Y, Hara H and Okamoto Y 1969 *Phys. Rev. B* **4**, 1950
[2] Toyoda E, Sakai A, Nakatsuka O, Isogai H, Senda T, Izunome K, Omote K and Zaima S 2008 The 5th International Symposium on Advanced Science and Technology of Silicon Materials, Nov. 10-14, Kona, Hawaii, USA
[3] Akatsu T, Scholz R and Gösele U 2004 *Journal of Materials Science* **39**, 3031
[4] Schroer E, Hopfe S, Werner P and Gösele U 1997 *Appl. Phys. Lett.* **70**, 327
[5] Reiche M 2008 *Materials Science Forum* **590**, 57, also online at http://www.scientific.net
[6] Ahn K-Y, Stengl R, Tan TY, Gösele U and Smith P 1989 *J. Appl. Phys.* **65**, 561
[7] Zhieng Yu, Muller DA and Silcox J 2004 *Journal of Applied Physics* **95**, 3362