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MECHANICAL DELAMINATION FOR THE MATERIALS INTEGRATION

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ABSTRACT

Mechanical exfoliation process in hydrogen implanted and bonded Si and the mechanical delamination method have been studied. Thin Si layers have been transferred from the temporary holder wafers to sapphire wafers using controlled direct wafer bonding and mechanical debonding. Helium ion-channeling measurements show that the silicon film on sapphire is crystalline in nature. We have also investigated the effect of the surface roughness on the bond strenght in SiO_2 to SiO_2 direct bonding. Prior to the bonding the wafer surfaces were modified using CHF_3 plasma etching. With a combination of surface roughening and activation treatments the surface energy of the bonded interface can be tailored to 500-1200 mJ/m^2 at temperature range of 200-1100°C. We suggest that controlled wafer bonding in combination with the delamination techniques could enable the fabrication of multilayer substrates and 3D-devices.

INTRODUCTION

Wafer bonding and various layer transfer techniques such as the ion-cutting of Si are becoming important processes in the microelectronics and microsystems industry [1]. Layer transfer by hydrogen implantation and wafer bonding can be realized by mechanical or thermal means [2,3]. Mechanical splitting of the implanted and bonded wafer pair can be performed under 300°C, whereas the thermal exfoliation process typically requires annealing at > 300°C [4, 2]. Conventionally, wafer-bonding research has targeted to obtain strongest possible bonds by using high temperature annealing step or plasma exposure or other surface activation procedure [1]. However, temporary bonded wafer pairs with intermediate bond strength of 500-1500 mJ/m^2 would be desirable in some applications. For example, the crystalline damage due to the hydrogen implantation in ion-cut Si layers could be removed by high temperature annealing before transferring the Si film onto temperature sensitive substrates. The controlled wafer bonding would also permit the mechanical delamination and transfer of processed thin layers to another substrate. To fully utilize the potential of this method, a bonding process yielding intermediate surface energy even after high temperature processing should be found. The mechanical transfer process itself could be performed near or at room temperature without generating excessive stresses due to the difference in thermal expansion coefficient. Fully processed wafers can be thinned down to 20-150 μm using mechanical grinding and etching. However, controlled wafer bonding in combination with ion-cutting could result in wafer scale processed thin films in the thickness range of 0.1-1.5 μm. In principle the delamination of the temporary bonded and processed layers would enable fabrication of integrated materials and 3-dimensionally packed devices.

The objective of this work was to study the mechanical exfoliation process in hydrogen implanted Si and to survey the possibility of transferring thin layers of Si onto another substrate using the controlled wafer bonding and the mechanical delamination methods. In order to form a mechanically debondable substrate we modified the surface topography of the wafers prior to the bonding. The influence of surface roughness on the bonding behaviour was evaluated by measuring the bond strength and the voids at the bonded interface.

EXPERIMENTAL

In the experiments, <100> oriented p-type Czochralski grown silicon and r-cut <1120> oriented sapphire wafers with a diameter of 100 mm were used. The resistivity of the Si wafers was 1-10 ohm-cm. A thermal wet oxide layer with a thickness of 500 nm was grown at 1050°C on some of the Si wafers to be used for hydrophilic Si to SiO_2 bonding (oxide/Si bonds) and SiO_2 to SiO_2 bonding (oxide/oxide bonds). Plasma enhanced CVD-silicon oxide layer was deposited on sapphire wafers to suppress the formation of interfacial voids during the bonding. Prior to the bonding the deposited oxide layer was smoothened with chemical-mechanical polishing (CMP).

For the implantation induced exfoliation experiments, the donor Si wafers were implanted with H_2^+ at 100 keV to a dose of 4.5 x 10^{16} H_2 / cm^2. Some of the donor wafers were also implanted with B^+ at 175 keV with a dose of 3 x 10^{15} B/cm^2 either before or after the hydrogen implantation. No thermal annealing was used to activate the implanted boron. The implanted donor wafers were subsequently bonded to oxidized handle Si wafers using plasma enhanced bonding process resulting in a surface energy of >2000 mJ/m^2 at 200°C [5]. The implanted and bonded wafer pairs were annealed in two steps: first 2 hours at 100°C and then 2 hours at 180-400°C. After annealing the surface energy of the H-implanted layer was measured using the crack opening method in air [1].

In order to form a substrate, which could be debonded even after high temperature processing, we studied the influence of the surface roughness on the bond strength of oxide/oxide bonds. The thermal oxide surfaces were modified (roughened) before the bonding using CHF_3 plasma etching in parallel plate RIE-reactor (Electrotech). During etching the plasma power was 100-250 W, and the etching time varied from 5s to 1 min. After the roughening treatments the wafers were cleaned in a SC-1 (NH_3:H_2O_2:H_2O) solution and DI-water. A short oxygen plasma exposure and subsequent DI-water cleaning was used for the activation of the surfaces prior to the bonding. Bonding was performed in air, and subsequently the wafer pairs were annealed at 200-1100°C for 2 hours.

The surface energies of the bonded interface and the H-implanted layer were measured from sliced rectangular samples using the crack opening method [1]. Debonding and layer transfer experiments were carried out by inserting a thin blade between the bonded wafers for the initiation of the crack. The bonded wafer pairs were

inspected for interfacial voids using IR transmission imaging and scanning acoustic microscopy (Sonix UHR2000). The surface roughness of the samples was measured with a Digital Instrument D3100 atomic force microscope (AFM) using silicon tips in the tapping mode. The crystalline quality of the thin Si film on sapphire was evaluated by using 2 MeV ^{4}He ions with channeled Rutherford backscattering method. The scattering angle was 160° in the channeling measurements.

RESULTS AND DISCUSSION

Figure 1 shows the surface energy of the bonded interface and the H-implanted layer. The wafers were exposed to oxygen plasma prior to the bonding. The basic condition for the exfoliation process is that the bonded interface is stronger than the hydrogen implanted layer. The surface energy of 2500 mJ/m^2 can be obtained for the thermal oxide to hydrophilic Si bonds at 200-250°C if a proper plasma activation procedure prior to the bonding is used. When a certain critical temperature is exceeded the strength of the H-implanted layer drops below 2500 mJ/m^2 enabling mechanical layer transfer, whereas thermal exfoliation calls for somewhat higher temperatures. The temperature required for exfoliation depends on the crystal orientation, boron doping level and implanted hydrogen dose. Boron doping and implantation prior to the hydrogen implantation reduce the strength of the H-implanted layer. Similar lowering in exfoliation temperature is observed in B-implanted but not activated and B-doped epitaxially grown layers. If boron is implanted after hydrogen, higher temperatures are needed for the exfoliation (Fig. 1). In order to determine the effects of boron doping in exfoliation, elastic recoil detection analysis (ERDA) and ion channeling measurements are underway in our laboratory. Thickness of the mechanically transferred Si layer is predetermined by the damage zone induced by the implantation. As the wafers are delaminated the cleavage occurs through the damage zone yielding a very uniform and smooth Si layer on top of handle wafer. Total thickness variation of the mechanically transferred layer given by optical reflectometry is typically around 2 nm. AFM-measurements yield a surface roughness of around 3 nm for the transferred Si layer.

The key to the mechanical layer transfer is good control of the bond strength. If we fabricate a SOI-structure with intermediate bond strength of 500-1500 mJ/m^2, the SOI-layer could be transferred to another substrate using mechanically induced delamination. To test this idea of controlled wafer bonding and subsequent mechanical separation of wafers we bonded thermally oxidized Si to unoxidized Si wafers at 800°C resulting in a bond strength of about 1000 mJ/m^2. The wafer pairs were thinned to a SOI-layer thickness of 15 μm using grinding and polishing processes. The finished SOI-structures were bonded either to oxidized Si or sapphire wafer using low temperature oxygen plasma assisted bonding yielding a surface energy of >1500 mJ/m^2 [5]. Subsequently, the wafer pairs were separated using a thin blade for the crack initiation. Since the surface energy of the first bonded interface was lower than that of the second, thick SOI-layers were transferred from the temporary holder wafer to another substrate (Fig. 2a). We also prepared thin film SOI-structures using thermal ion-cutting process.

The exfoliation of the bonded wafer pairs was carried out at 500°C. After splitting the ion-cut SOI-wafers were polished and bonded to oxidized Si or sapphire wafers. Also in this case the bonding was carried out using the plasma-assisted process. The silicon-on-sapphire (SOS) wafer with a transferred thin Si-layer is presented in Fig. 2b. With normal thermal ion-cutting the fabrication of silicon-on-sapphire structures is difficult because of the large difference in thermal expansion coefficient [1]. However, in our experiments the bond between the debondable SOI-substrate and sapphire was formed at 120°C without generating excessive stresses. Figure 3 shows the $^4He^+$ ion-channeling spectrum of the thin film SOS-structure. For comparison a spectrum in random condition and a spectrum of a virgin sample are also presented. The ion-channeling results suggest that there is a very low amount of lattice damage in the Si layer. This damage due to the hydrogen implantation could be removed by annealing at high temperature. Figure 4 shows the AFM-image of the transferred SOS-layer. The measured surface roughness was around 0.1 nm indicating than no polishing is needed after separation of the wafers.

Our experiments show that both thin and thick Si films can be transferred onto another substrate using controlled wafer bonding and mechanical delamination. It appears that during the mechanical separation of wafers the crack propagates along the weakest interface without derailing to other interfaces provided that there is a sufficient difference in the interface strengths. To fully utilize the technique, it is necessary to find bonding processes for engineering or limiting the bond strength to prescribed level. The initial bonding between hydrophilic Si wafer surfaces is caused by hydrogen bonds [1]. Because of the short-range nature of hydrogen bonds, the bonding is sensitive to surface roughness. In order to control the bond strength we modified the surface topography of the thermally oxidized Si wafers using plasma etching. Figure 5 shows AFM images for untreated reference SiO_2 and plasma etched SiO_2. Plasma etching in RIE-mode induced texture with 30-100 nm wavelengths on the surface. The influence of the surface roughness on the bond strength in SiO_2 to SiO_2 bonding is illustrated in Fig. 6. The wafers were cleaned with SC-1 solution just before the bonding in air, and no plasma activation was used. After annealing at 1100°C a surface energy of 700 mJ/m^2 is measured for the samples etched in CHF_3 plasma prior to the bonding as compared to the strength of > 2000 mJ/m^2, which was obtained for the reference sample. The result is consistent with those reported by Roberts [6]. No macroscopic voids were detected for the plasma-etched samples. This kind of approach could be used to make debondable substrates with mechanically thinned SOI-layers. However, the strength of the bonded interface is too low for preparing ion-cut thin SOI-layers, since thermal exfoliation at 300-600°C requires surface energy of about 500 mJ/m^2 [2].

To further adjust the bonding energy we exposed the roughened surfaces to oxygen plasma before the bonding. The measured surface energies for the etched and plasma activated samples are depicted in Fig. 7. If plasma activation is used strong bonding is obtained at 200°C even though the SiO_2 surfaces were pre-roughened by dry-etching. This could be caused by the decrease in surface roughness due to the short oxygen plasma exposure. We tried to verify this with AFM, but were not able to obtain reliable roughness values probably because of plasma induced surface charging. High bond strength obtained for pre-roughened activated samples could also result from the increase in the initial bonding force leading to the enhanced formation of Si-O-Si covalent bonds. When smooth (non-etched) plasma activated SiO_2 is bonded to dry-

etched non-activated SiO_2 the surface energy is between 500 and 1200 mJ/m^2 in the temperature range of 200-1100°C. This surface energy range enables thermal ion-cutting as well as the debonding after high temperature annealing. Scanning acoustic microscopy measurements showed that there are no macroscopic voids in the samples (Fig. 8). We believe that plasma exposure increases the number of OH-groups resulting in increased surface energy at low temperatures. This is supported by the contact angle measurements, which were carried out after plasma exposure. Plasma-activated and DI-water cleaned SiO_2 surfaces showed a very hydrophilic behaviour, and the contact angle of DI-water dropplet on surface was so low (<5°) that it could not be measured. As the wafers are contacted the area over which the bonding really occurs is limited when the surfaces are rough. We believe that this limits the surface energy to 1500 mJ/m^2 even at high temperatures.

Based on the results, we suggest that with well-adjusted surface modification and activation treatments one could achieve useful debondable substrates. We used a thin solid wedge for the delamination of the wafers but more reliable delamination methods such as gas or fluid wedges may also be used [3]. In addition, the other surface of the bonded wafer pair could be clamped to a vacuum chuck for better cleaving action if one of the wafers is fragile.

CONCLUSIONS

When the bonded wafers are separated with mechanical splitting forces, the crack propagates through the weakest interface. Our result show that controlled wafer bonding and mechanical delamination method enable near room temperature fabrication of integrated substrates such as silicon-on-sapphire (SOS). Surface topography modification is an effective approach to control the bond strength in direct wafer bonding. We suggest than controlled direct wafer bonding can be used for the fabrication of debondable substrates for 3D-integrated devices.

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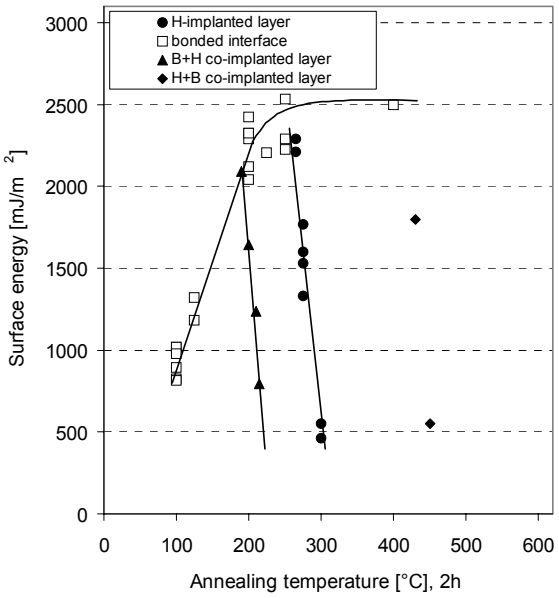

Fig. 1 Measured surface energies for the bonded interface, H-implanted layer and the boron and hydrogen co-implanted layer. Hydrogen was implanted with H_2^+ at 100 keV with a dose of 4.5 x 10^{16} cm^{-2}. Boron was implanted with B^+ at 175 keV with a dose of 3 x 10^{15} cm^{-2} either before (B+H) or after the hydrogen implantation (H+B). The annealing time was 2 hours.

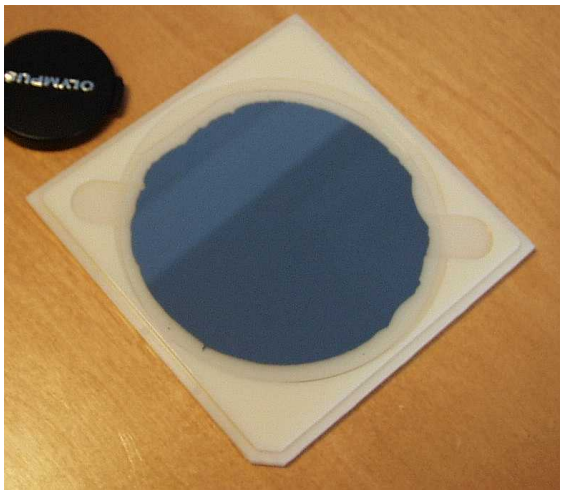

Fig. 2a Photograph of 100 mm thick film SOS-wafer fabricated by mechanically releasing the SOI-layer from temporary substrate to a sapphire wafer. Thickness of the Si film is 15 µm.

Fig. 2b Photograph of 100 mm thin film SOS-wafer fabricated by mechanically releasing the SOI-layer from temporary substrate to a sapphire wafer. Thickness of the Si film is 410 nm.

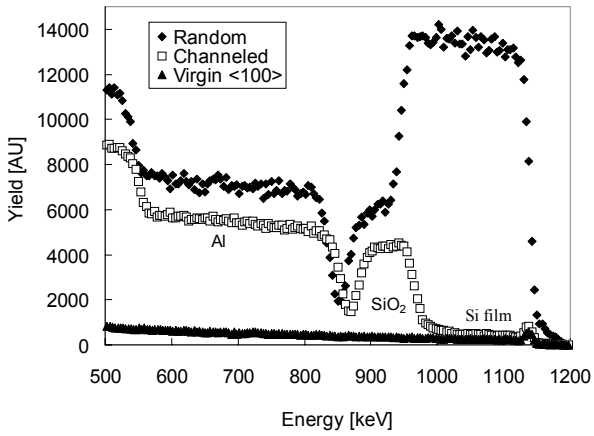

Fig.3 RBS-channeling spectrum of the transferred thin Si layer on top of sapphire substrate. The random and virgin spectra are shown as a reference. Silicon film and intermediate SiO_2 layer thicknesses given by scattering measurement are 410 and 130 nm respectively.

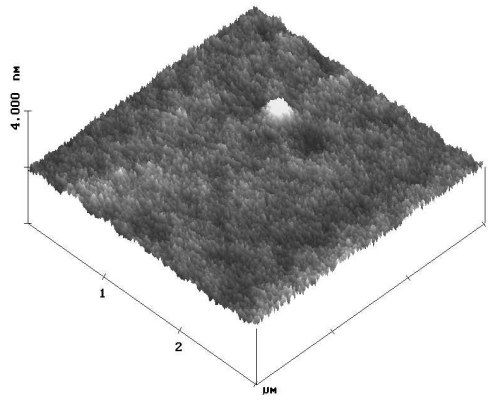

Fig. 4 AFM image of debonded and transferred thin Si layer on a sapphire wafer. The surface roughness is about 0.1 nm.

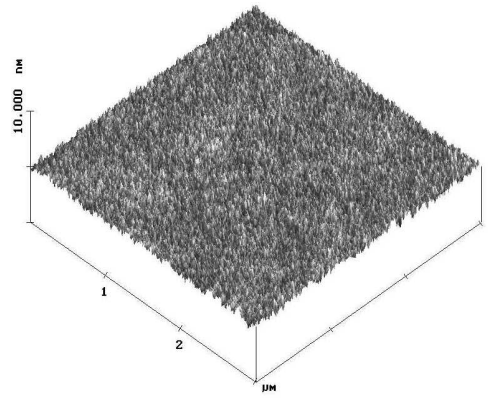

Fig. 5a AFM-image of as-grown thermal SiO_2 surface. The RMS- roughness is ~ 0.2 nm.

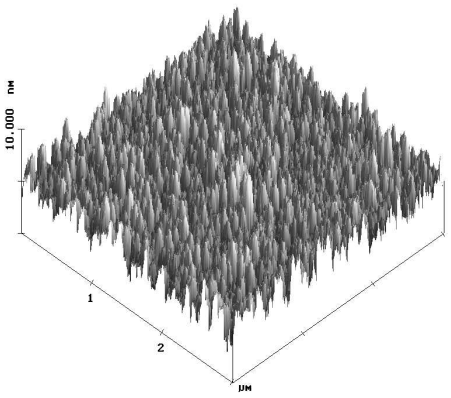

Fig. 5b AFM-image of SiO_2 surface etched in CHF_3 plasma for 30s. The RMS-roughness is 0.8 nm.

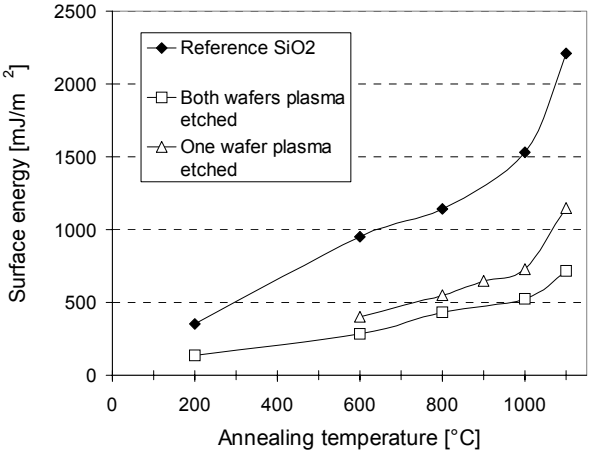

Fig. 6 Measured surface energies for SiO_2 to SiO_2 bonds. The surfaces were etched for 30s in CHF_3 plasma prior to the bonding. The untreated reference sample is shown for comparison. The bond annealing time was 2 hours.

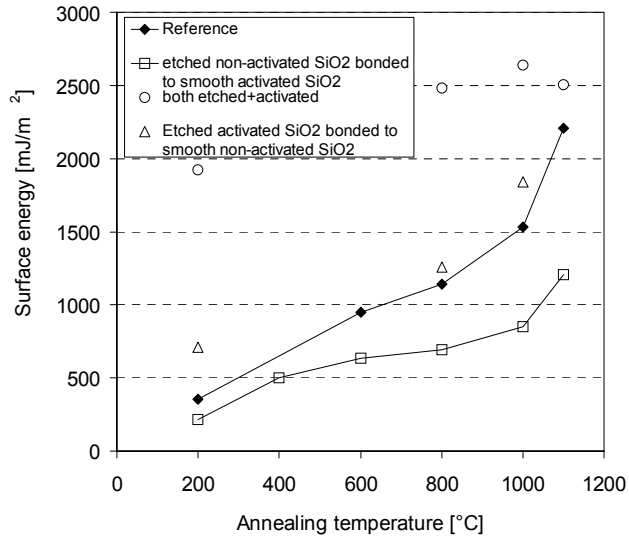

Fig. 7 Measured surface energies for the plasma etched and activated SiO_2 to SiO_2 bonds. The CHF_3 plasma was used for the surface etching. Short oxygen plasma exposure was used for the surface activation. The untreated reference sample is shown for comparison. The bond annealing time was 2 hours.

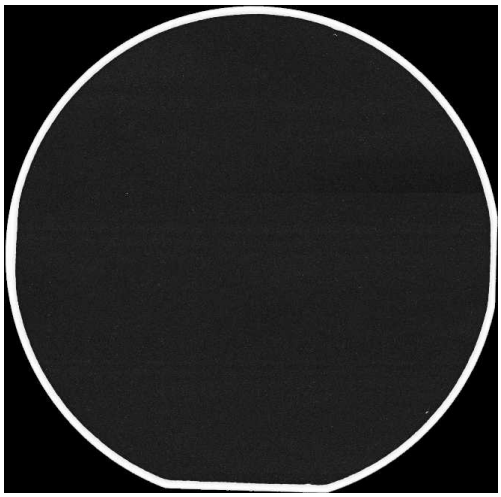

Fig. 8 Scanning acoustic misroscopy image of debondable SiO_2 to SiO_2 bond. The one wafer was modified in CHF_3 plasma before the bonding. The other wafer was exposed to oxygen plasma prior to the bonding. After bonding the wafer pair was annealed at 600°C.