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Effects of surface activation time on Si-Si direct wafer bonding at room temperature

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Abstract

Surface activated bonding (SAB) based on argon ion beam irradiation was used to directly bond Si and Si wafers at room temperature, and the effects of the surface activation time on the Si-Si bonding were investigated. The experimental results show that the surface activation treatment with a proper duration is beneficial to the reduction of surface roughness of Si wafers and the realization of high bonding strength. The Si-Si wafers bonded after the surface activation of 420 s has an extremely low percentage of area covered by voids (0.08%) and a high bonding strength (9.45 MPa). Meanwhile, the annealing at 500 °C does not lead to a significant change in the percentage of area covered by voids for Si-Si bonding. Besides, the transmission electron microscope characterization indicates that the argon ion beam irradiation of 180 s can result in the formation of an amorphous Si layer with a thickness of approximately 10.6 nm at the Si-Si bonding interface, and the whole cross-section structure of the Si-Si bonding consists of a Si substrate, an amorphous Si layer and a Si substrate.

1. Introduction

Wafer bonding is one of the key technologies of integrating homogenous or heterogeneous semiconductor materials or devices. In recent years, it has been extensively studied and used in a lot of fields, such as radio frequency and power electronic device fabrication, micro-electro-mechanical system (MEMS) packaging, system-in-package, and 2.5D/3D integration [1–8]. The conventional bonding method usually needs high-temperature treatment, which will lead to a residual thermal stress at the bonding interface due to the thermal expansion coefficient mismatch between two heterogeneous materials, and thereby degrade the performances of devices [9]. Many researchers have reported the bonding methods with heat treatment. Shi et al prepared GaAs-based n-n and p-n interface junctions by wafer bonding at 400 °C and subsequent high-temperature annealing under N₂ flow at 600 °C [10]. Tanabe et al achieved GaAs-InGaAs heterostructure by wafer bonding and annealing at 380 °C [11]. At present, the main methods to solve this problem are surface activated bonding (SAB) and metal interlayer bonding. However, the latter will introduce a metal layer at the bonding interface, which has an adverse effect on the heat dissipation of the devices.

SAB can realize the wafer bonding without a metal interlayer at low or even room temperature, neglecting the mismatches between heterogeneous materials in thermal expansion coefficient [12–14] and lattice constant. A variety of heterogeneous materials, such as GaN-Si [15], GaAs-Si [16–18], diamond-Si [19–21], GaSb-Si [22], and GaN-diamond [23, 24], have been integrated together by SAB. The key point of this bonding method is the surface activation with ion or fast atom beam irradiation, which can clean and activate the surfaces. After the surface activation, two wafers are immediately bonded together in ultra-high vacuum. However, the surface activation can cause the lattice damage of wafer surfaces, which results in the formation of an amorphous layer at the bonding interface [25, 26]. This amorphous layer can enhance the diffusion of surface atoms and reduce the...
activation energy for the formation of interatomic bonds, so it plays an important role in wafer bonding [10]. Mu et al. realized the GaN-SiC bonding based on Ar ion beam surface activation at room temperature and got a bonding interface with about 3.6 nm amorphous layer [27]. In terms of interfacial voids, Morishita et al. used Ar fast atom beam to activate the Si wafer surfaces, and obtained the bonded Si-Si specimen with a low percentage of area covered by voids (about 1.0%) [28].

As mentioned above, SAB technique has made a lot of research progress. However, the investigations on the optimization of SAB process parameters are still very insufficient. Therefore, in this work the effects of the surface activation time on the bonding strength and percentage of area covered by voids for the Si-Si direct bonding at room temperature were investigated, and the thermal stability of the bonded Si-Si wafers was explored.

2. Experimental methods

Si (100) wafers with a profile dimension of 10 mm × 11 mm × 0.5 mm were used in the SAB experiment. First of all, acetone, absolute ethanol and deionized water were used to clean the Si wafers in sequence, which can preliminarily remove dust particles and organic contaminants. Secondly, for the Si-Si bonding two cleaned Si wafers were fixed on the upper and lower specimen holders in the vacuum chamber of the bonding equipment which was vacuumed to 3.0 × 10⁻⁵ Pa, after that the surfaces of these two Si wafers were activated simultaneously by Ar ion beam irradiation under the beam parameters of a 55 mA current, a 260 V acceleration voltage and a 45° incident angle. This step can further remove the residual contaminants and the intrinsic oxides and produce dangling bonds on the surfaces of the Si wafers. Thirdly, the lower specimen holder moved upwards to make these two Si wafers keep in full contact with each other for 300 s, which produced the bonded Si-Si wafers. In order to investigate the surface activation time dependence of the Si-Si bonding quality, different activation times were chosen, including 60 s, 180 s, 300 s, 420 s and 540 s. Figure 1 shows the process flow of the Si-Si surface activated bonding. Finally, the bonded Si-Si specimens were annealed in Ar atmosphere at 500 °C for 600 s with a temperature rising rate of 5 °C s⁻¹ for the evaluation of the thermal stability of the Si-Si wafers bonded by SAB.

Atomic force microscopy (AFM) was used to measure the surface morphologies and root-mean-square (RMS) surface roughnesses of the original and activated Si wafers. To estimate the bonding qualities of the
bonded specimens, the percentages of area covered by voids were evaluated by scanning acoustic microscope (SAM), and the bonding strengths were measured with a universal testing machine. Additionally, the bonding interface of a bonded specimen was analyzed by transmission electron microscopy (TEM) and energy dispersive spectrometer (EDS).

3. Results and discussion

3.1. Morphology characterization

Figures 2 and 3 show the AFM images of the original and the activated Si wafers, and the variation curve of the RMS surface roughness of activated Si wafer with activation time, respectively. It can be seen that the RMS surface roughness presents a variation tendency of first decreasing and then increasing with the increase of activation time from 60 s to 540 s. The RMS surface roughness reaches a minimum value (0.34 nm) at 420 s, and the surface roughness of every activated Si wafer is lower than that of the original Si wafer.

This phenomenon is connected with the existence of nano scale protrusions and pits on the Si wafer surface. When the activation time is less than 420 s, the protrusions are etched more strongly by Ar ion beam than the
pits. The protrusions are etched lower and lower when the activation time increases gradually, so the surface roughness decreases. However, when the activation time is more than 420 s, the number and height of protrusions have been significantly reduced. Therefore, the ion beam etching on pits can not be neglected. Consequently, the surface roughness begins to increase as the pits are etched deeper and deeper. This result indicates that a proper surface activation time can reduce the surface roughness of Si wafer, which is conducive to the Si-Si bonding.

3.2. Interfacial void analysis
The percentages of area covered by voids of five bonded specimens with the different activation times were analyzed by SAM. Figure 4 shows the SAM images, from which the number, size and distribution of the voids at the bonding interfaces can be observed. The percentages of area covered by voids of the bonded specimens before and after annealing are listed in table 1. These five specimens all have low percentages of area covered by voids, especially for the one with an activation time of 420 s, which has an extra-low value of about 0.08%. The voids in these specimens may be caused by particulate contaminants on the surfaces of the Si wafers. Besides, these five bonded specimens were annealed at 500 °C for 600 s in Ar atmosphere. The SAM images and percentages of area covered by voids of the annealed specimens are shown in figure 5 and listed in table 1, respectively.

The 60 s-activation specimen had an increased void number after the annealing compared with that before the annealing, and its percentage of area covered by voids changed from 0.18% to 0.36%. There was still only one void for the 180 s-activation specimen after the annealing, and the size of the void was almost unchanged. The number of voids did not change for the 300 s-activation specimen after the annealing, but one of the voids became smaller than before, so the percentage of area covered by voids decreased. The void completely disappeared for the 420 s-activation specimen after the annealing, and the percentage of area covered by voids reduced to zero. The percentage of area covered by voids of 540 s-activation specimen was still 2.0%, which was almost unchanged after the annealing. On the whole, the percentages of area covered by voids of these specimens did not increase after the annealing except the 60 s-activation one, which means that the bonded Si-Si wafers have a good thermal stability. Under the bonding parameters as mentioned above, the 60 s-activation time is not enough for achieving adequate surface activation, so its thermal stability is not so good as the others.

3.3. Bonding strength testing
The bonding strengths of the bonded Si-Si specimens were evaluated by tensile strength test. A pair of Si wafers of every bonded specimen were fixed to the upper and lower specimen holders of a universal testing machine with a high-adhesive-strength resin glue, and a tensile force perpendicular to the bonding interface was applied to the bonded specimen. The applied tensile force gradually increased from 0 N until the specimen was broken to two parts. The bonding strengths of the bonded specimens are shown in table 2. Overall, with the activation time increasing from 60 s to 540 s, the bonding strength first increases and then decreases, reaching a peak value of 9.45 MPa at 420 s. The prolongation of the surface activation time from 60 s to 420 s causes the decrease of surface roughness and the increase of surface dangling bonds, which jointly make the bonding strength rise. However, with the increasing of the surface activation time from 420 s to 540 s, the bonding strength decreases.
The main reason for that may be attributed to the thicker amorphous Si layer between the two bonded Si wafers with the activation time of 540 s. The amorphous Si layer is formed at the bonding interface due to the Ar ion beam irradiation. The thickness of the amorphous Si layer becomes larger when the activation time increases, so the 540 s-activation specimen has the thickest amorphous Si layer. But the amorphous Si layer has high-density defects with a poor strength, which results in a decrease in the bonding strength.

Figure 6 shows two fractured surfaces of the 420 s-activation specimen after the bonding strength testing. As can be seen, the fracture of the region I occurred at the bonding interface. However, the fracture of the region II occurred at the position of the adhesive glue used to adhere the specimen to the holder, and the fracture in the other regions occurred at the bulk Si. The region I accounts for only a small area proportion of about 9%. Therefore, the fracture of the bonded specimen mainly occurred at the adhesive glue and bulk Si, which means that the bonding strength is close to the mechanical strength of the solidified resin glue or bulk Si. The high Si-Si

| Activation time (s) | Percentage of area covered by voids (%) | Before annealing | After annealing |
|---------------------|-----------------------------------------|------------------|-----------------|
| 60                  | 0.18                                    | 0.36             |
| 180                 | 0.10                                    | 0.10             |
| 300                 | 0.36                                    | 0.10             |
| 420                 | 0.08                                    | 0                |
| 540                 | 2.0                                     | 2.0              |

Table 1. Percentages of area covered by voids of the bonded Si-Si specimens with the different activation times before and after annealing.
bonding strength was attributed to the proper surface activation, which leaded to the low surface roughnesses of the two Si wafers and the high-density dangling bonds on the Si wafer surfaces so that the two Si wafers could be bonded together tightly by strong interatomic bonds.

3.4. TEM observation and EDS analyses of the bonding interface

To explore the interfacial microstructure of the bonded Si-Si wafers, TEM was used to observe the bonding interface of the 180 s-activation specimen. Figure 7(a) is a low magnification image (2 × 10^4 times), which shows a clear bonding interface without any voids between the two Si wafers. Figure 7(b) is a high magnification image (5 × 10^5 times) of the bonding interface. It can be seen that there is an amorphous Si layer with a thickness of approximately 10.6 nm between the two Si wafers, which indicates that the bonded specimen has a three-layer structure consisting of a crystalline Si, an amorphous Si layer and a crystalline Si. The formation of the amorphous Si layer was caused by Ar ion beam irradiation during the surface activation. The injection of energetic Ar ions destroyed the Si crystal structure, resulting in the amorphization of the Si wafer surfaces.

Figure 8 shows the EDS mapping images of the Si-Si bonding interface. The distributions of Ar and Si elements are marked in yellow and red, respectively. It can be seen that Ar element is mainly distributed in the amorphous Si layer at the bonding interface, and there is also a tiny amount of Ar element distributing in the

| Activation time (s) | Bonding strength (MPa) |
|---------------------|------------------------|
| 60                  | 1.25                   |
| 180                 | 4.18                   |
| 300                 | 8.90                   |
| 420                 | 9.45                   |
| 540                 | 3.70                   |
Figure 6. Surface profiles of the two fractured parts after the tensile strength test: (a) the part A and (b) the part B.

Figure 7. (a) Low-magnification TEM image ($2 \times 10^4$ times) of the Si-Si bonding interface with two illustrations showing the diffraction patterns of the two Si wafers; (b) high-magnification TEM image ($5 \times 10^5$ times) of the Si-Si bonding interface.

Figure 8. (a) High-resolution ($2.5 \times 10^5$ times) TEM image of the Si-Si bonding interface, and (b) its high-resolution EDS mapping images: Ar and Si elements are marked yellow, and red colors, respectively.
crystalline Si, which is also caused by the surface activation. During the activation process, some Ar ions were injected into the amorphous Si layer and even the crystalline Si.

4. Conclusions

In addition to removing the residual contaminants and oxides and producing dangling bonds on Si wafer surfaces, the surface activation treatment with a proper length of time can also reduce the surface roughness of Si wafer, all of which are beneficial to the wafer bonding. High quality Si-Si direct bonding was successfully realized by SAB at room temperature. The 420 s-activation Si-Si bonding specimen had a 0.08% percentage of area covered by voids which changed to zero after the annealing at 500 °C and a bonding strength of 9.45 MPa. The bonded Si-Si wafers with an activation time of 180 s had a three-layer structure consisting of a crystalline Si, a 10.6 nm-thickness amorphous Si layer and a crystalline Si.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflict of interest

The authors declare that they have no conflict of interest.

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