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Research on the Effects of Process Parameters on Surface Roughness in Wet-Activated Silicon Direct Bonding Base on Orthogonal Experiments

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Surface roughness is a very important index in silicon direct bonding and it is affected by processing parameters in the wet-activated process. These parameters include the concentration of activation solution, holding time and treatment temperature. The effects of these parameters were investigated by means of orthogonal experiments. In order to analyze the wafer roughness more accurately, the bear ratio of the surface was used as the evaluation index. From the results of the experiments, it could be concluded that the concentration of the activation solution affected the roughness directly and the higher the concentration, the lower the roughness. Holding time did not affect the roughness as acutely as that of the concentration, but a reduced activation time decreased the roughness perceptibly. It was also discovered that the treatment temperature had a weak correlation with the surface roughness. Based on these conclusions, the parameters of concentration, temperature and holding time were optimized respectively as $NH_4OH:H_2O_2=1:1$ (without water), 70 °C and 5 min. The results of bonding experiments proved the validity of the conclusions of orthogonal experiments.

Keywords: wafer bonding, surface hydrophilicity, wet activation, process optimization.

1. INTRODUCTION

Silicon wafer bonding technology is commonly used in the fabrication and packaging of silicon-on-insulator substrates, microelectronic devices, power electronic devices, micromechanics devices, optoelectronic devices, and three-dimensional microelectromechanical systems [1 – 3]. Many techniques have been developed to realize wafer bonding including fusion bonding, hot-pressing bonding and adhesive bonding. Among these techniques, direct bonding has garnered the most attention due to its uniqueness. In direct bonding, no external force or electrical field is needed. Thus it is regarded as a very promising micromachining technique in semiconductor and micromachining industry.

In direct bonding process, surface activation is a pivotal step for both hydrophilic and hydrophobic bonding. Because of its relatively convenient and economical process, wet surface activation is widely used. In this technique, the surfaces of silicon wafers are treated with several kinds of chemical solutions before bonding. These treatments clean the wafer surfaces on which high-density hydroxyls are then absorbed. These hydroxyls can be linked to water molecules by hydrogen bonding. Water molecules can also be linked to each other through the hydrogen bonding. Thus two wafers can be bonded to each other via water molecules which are anchored to the hydroxyls absorbed on wafer surfaces [4 – 6]. As shown in Fig. 1, the linkage of three water molecules can form a bridge across the two bonded surfaces. Since the distance between two hydrogen bonded oxygen atoms is 0.276 nm, the length of the linkage bridge is three times that, or 0.828 nm, i.e. silicon wafers with surface roughness over 0.4 nm can be bonded. In a word, the purpose of surface activation is to improve the hydrophilicity of wafer surface.

The validity of wet surface activation has been proved in experiments and productions for hydrophilicity improvement. However, it is for a long time that the process parameters of wet surface activation are decided empirically. The effects of processing parameters are still unclear [7 – 9]. It is very difficult to obtain an optimized surface activation process by empirical methods. Thus the shortcomings of empirical methods must be eliminated by the use of scientifically valid models which provide a means of establishing the optimum process parameters, as well as facilitating the real-time control of the manufacturing process.

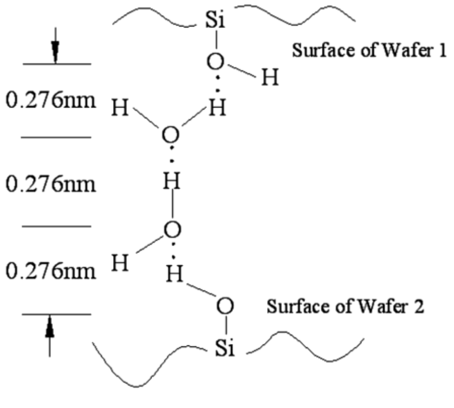

Fig. 1. Schematic illustration of a linkage of three water molecules between two hydrophilic mating surfaces to bridge a gap between wafers

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2. EXPERIMENTAL EVALUATION INDEX

In order to unveil the effects of the process parameters effectively, a series of experiments were designed and performed. It is well known that *RMS* (Root Mean Square) was widely accepted as a critical index of surface roughness in wafer direct bonding [10, 11]. However, *RMS* is not the best choice for roughness evaluation in bonding process because it only concerns the absolute value of the height of surface roughness. In other words, the roles of wave crest and the trough are regarded equally in *RMS* measurement.

According to the normal procedure of *RMS* measurement, five areas on a wafer are inspected as the sampling windows. Among those areas, one is located in the center of the wafer. The other four areas are arranged averagely as a ring array around the wafer center and the radius of the ring array is half of the wafer radius as shown in Fig. 2.

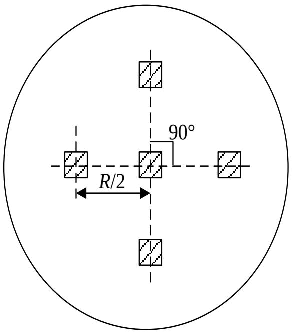
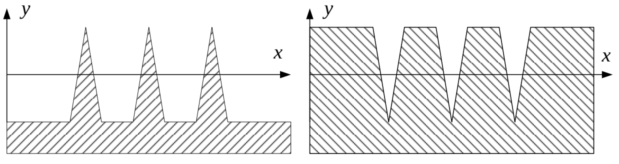


Fig. 2. The locations of measuring areas on silicon wafer

The nano-scale profile of the sampling area is measured by AFM (Atomic Force Microscope) and then the *RMS* is calculated as the square root of the arithmetic mean of the squares of the heights of measuring points which are represented by y_i in Eq. (1). The *RMS* of whole wafer is the average of the values of all five sampling windows.

$$RMS = \sqrt{\frac{1}{n}\sum_{i=1}^{n} y_i^2} \,. \tag{1}$$

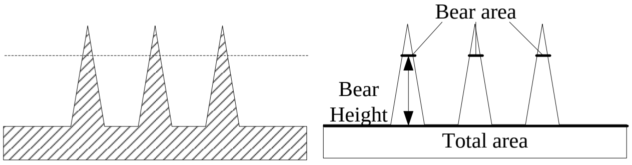
It must be admitted that *RMS* is a very important parameter for surface quality evaluation. But according to Eq. 1, the *RMS* values of the profiles shown in Fig. 3 a and Fig. 3 b are the same. That is to say, the convex and concave wafers have the same ability to be bonded. But for the direct wafer bonding, if perfect wafers, which have absolutely flat surface with zero roughness, are bonded to the two types of wafers respectively, it is obvious that convex one is much more difficult to be bonded than the other because of the less contact areas.



a b

Fig. 3. Two different types of surface profiles: a – convex; b – concave

Based on the explanation above, a more proper evaluation index of roughness is proposed: bear ratio. From Fig. 4, the definition of bear ratio can be understood. At first, a certain height, i.e., bear height, is decided before bear ratio measurement. Then a plane is made just at this height over the wafer surface. The crest is trimmed by this plane and the consequent area is named the bear area. The ratio between the bear area and total area is defined as the bear ratio. It is clear that for a constant bear ratio, the lower the bear height is, the better the roughness. And by the aid of AFM, Bear ratio can be obtained easily.



a b

Fig. 4. Definition of bear ratio: a – surface profile; b – bear ratio calculation

3. EXPERIMENTAL DESIGN

The procedure of wet surface activation consisted of several chemical solution treatments and 4 in silicon wafers with the crystal orientation of < 100 > were used as the test samples.

Wafers were cleaned by acetone and SPM (Sulfuric acid – hydrogen Peroxide Mixture).

Dipped in dilute hydrofluoric acid for 30 seconds to remove native oxide layer on the surface.

Dipped in RCA-1 solution (NH_4OH: H_2O_2: H_2O=1:1:5, volume ratio) for 15 min at the temperature of 75 °C.

And deionized water flushing was followed each processing. Then the wafers were spin-dried after rinsing. An infrared light was used to raise the temperature, thus the spin-drying time was reduced.

According to the activation procedures, it was obvious there were three important parameters, which were the volume ratio, holding time and treating temperature of RCA-1 solution respectively [12, 13]. In order to reduce the number of experiments, orthogonal method was used to design the experiment plan. Since there were three parameters to be investigated, the L_9 (3^4) orthogonal array was chosen to organize the experiments. The L_9 (3^4) meant there were nine experiments in total with three levels and four factors. Because only three parameters were investigated, in orthogonal array there was a void factor D, which did not exist and was only used as a verification factor in the result analysis. Every parameter had three levels as shown in Table 1 according to which orthogonal array was designed (Table 2)

Table 1. Factors and levels of experiments

Level	Factors (Parameters)		
	A:	B:	C:
	Volume Ratio (NH_4OH：H_2O_2：H_2O)	Time, min	Temperature, °C
①	1:1:5	5	25
②	1:1:2	15	50
③	1:1:0	30	70

Table 2. Orthogonal experiment array

No.	Factor			
	A	B	C	D
1	①	①	①	①
2	①	②	③	②
3	①	③	②	③
4	②	①	③	③
5	②	②	②	①
6	②	③	①	②
7	③	①	②	②
8	③	②	①	③
9	③	③	③	①

4. EXPERIMENTAL RESULTS

Based on the orthogonal experiment design above, nine groups of wet-activated processes were performed. Next the bear ratio of each sample was measured by AFM. The bear height h_{BR} was acquired as shown in Table 3. According to the measurement by AFM, the h_{BR} of untreated surface was 0.625 nm. But after the wet-activated process, the h_{BR} increased for all the samples. Therefore, the process should be optimized to alleviate the inconvenience of surface roughness.

Table 3. Results of bear height h_{BR}

No.	(NH_4OH : H_2O_2 : H_2O)	Time (min)	Temp. (°C)	Null	h_{BR} (nm)
1	①1:1:5	① 5	①25	①	1.217
2	①1:1:5	②15	③70	②	1.240
3	①1:1:5	③30	②50	③	1.357
4	②1:1:2	① 5	③70	③	1.055
5	②1:1:2	②15	②50	①	1.073
6	②1:1:2	③30	①25	②	1.110
7	③1:1:0	① 5	②50	②	0.976
8	③1:1:0	②15	①25	③	0.949
9	③1:1:0	③30	③70	①	1.057

5. DISCUSSION AND ANALYSIS

In order to analyze the results of orthogonal experiments, the intuitive analysis method was applied to reveal the effects of the different parameters. The average and range of each parameter are shown in Table 4 and it is obvious that:

a) The range of concentration was the largest among all three parameters. Thus it had the most distinct effect on the h_{BR}. And along with the increase of concentration, the h_{BR} became lower and lower. That was to say, higher concentration was desirable to achieve good surface roughness.

b) Although the range of holding time was not as high as that of concentration, it could not be neglected. According to the variation of range of holding time, the h_{BR} increased continuously as the time was prolonged. In other words, the longer the holding time was, the worse the surface roughness.

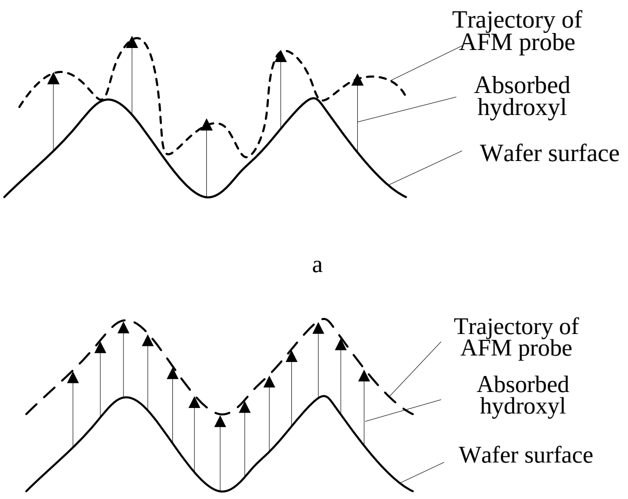
c) As far as the treat temperature was concerned, the range was lower relatively, meaning it had a weak effect on surface roughness.

And as to the null column in Table 3 (the factor D), the range was very minimal. As such it was clear that this parameter had no relationship with surface roughness. Because this parameter did not exist, the conclusion was realistic and it also proved that the results of orthogonal experiments were reliable.

Table 4. Results of intuitive analysis

		Concentration	Time	Temperature	Null
Average	①	1.271	1.083	1.092	1.116
	②	1.079	1.087	1.135	1.109
	③	0.994	1.175	1.117	1.120
Range		0.277	0.092	0.043	0.012

According to the experimental results and discussion above, it was proved that the higher concentration led to lower surface roughness. This conclusion could be explained based on the measuring principle of AFM. The roughness measured by AFM were acquired by the aid of nano-scale probes which contacted (or tapped) the actual profiles. After activated process, hydroxyls were absorbed on the top of wafer surface. Thus the trajectory of AFM probe was the envelope of hydroxyl top and silicon surface which was not covered by hydroxyl as shown in Fig. 5 a. When the higher concentration solution was used, the density of hydroxyl was raised due to more hydroxyls were absorbed. In this case, the probe trajectory was smoother as shown in Fig. 5 b and the value of roughness was lower. In fact, for as-activated surface, the real roughness of bare silicon wafer can not be measured because of the hydroxyl envelop. But since the wafer bonding is achieved by water linkage ended by hydroxyl as mentioned in the first section, the actual nanoscale surface profile of hydroxyl is the concerning object of roughness measurement. Therefore, a higher concentration could achieve better surface quality.



a

b

Fig. 5. The difference of roughness measurement by AFM of a – the surface with sparse absorbed hydroxyl; b – the surface with dense hydroxyl

As to the holding time, because the activating solution was weak alkaline, it would etch silicon laggardly. Thus the longer the holding time is, the higher the roughness.

Based on these conclusions described above, an optimized wet-activated process was designed:

1) Cleaning: Wafers were cleaned by acetone in supersonic cleaner for 20 minutes and then boiled in SPM (H_2SO_4:H_2O_2 = 2:1) for 20 minutes. This eliminated organic contaminations and metal particle contaminations. A following dipping in 10 % hydrofluoric acid for 30 seconds was conducted to remove native oxide layer.

2) Activation: The activation solution was prepared based on the conclusions mentioned above. The concentration was NH_4OH:H_2O_2 = 1:1. The holding time and treat temperature were 5 min and 70 °C respectively.

3) Bonding: Activated wafers were bonded together without any other aid method. Then bonded wafers pair were annealed at 300 °C for 3 hours to enhance the bonding strength.

The bonded wafers were inspected by infrared camera and the result is shown in Fig. 6. And according to the SEM image of cross section of bonding interface (Fig. 7.), the interface was smooth and even. The thickness was less than 1 μm. It was obvious that in the central part of bonded wafers, there was no prominent defect, like void and bubble which would induce Newton ring in infrared image, existing in bonding interface, i.e., high quality silicon direct bonding could be realized if the optimized wet-activated process was applied.

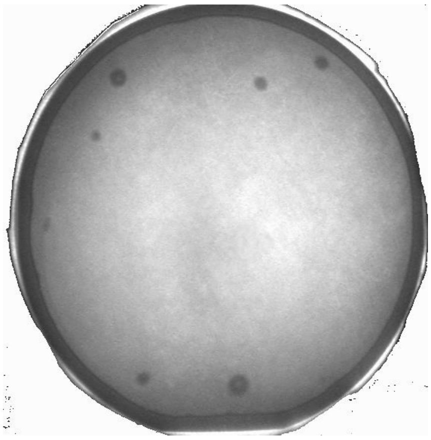


Fig. 6. Infrared inspection result of bonding wafers

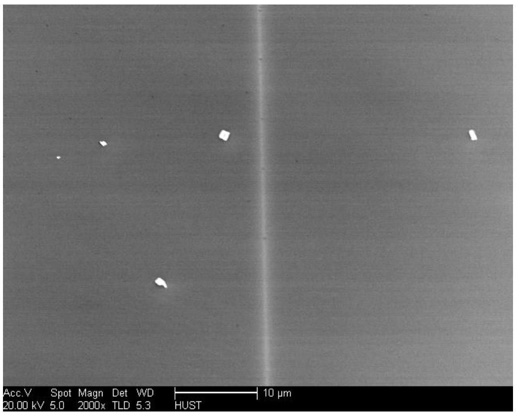


Fig. 7. Interface of bonding wafers

The difference between the optimized process and traditional one is shown in Table 5. The traditional activated process, namely RCA-1 or SC-1 cleaning process, was widely used in wafer direct bonding [12 – 14]. However, the effects of processing parameters and corresponding mechanisms were seldom investigated intentionally. Now along with the rapid development of semiconductor manufacturing technology, higher processing efficiency and better bonding quality were more and more desirable for academic and industrial applications. Thus the optimized process was developed based on the understanding of effects of processing parameters. From Table 5, it is obvious that the holding time was reduced sharply from 15 min to 5 min with the corresponding variety of concentration. Furthermore, the results of experimental analysis and theoretical explanation on those effects also offered a technical support by which researchers and engineers could modify their bonding processes more freely.

Table 5. Comparison between optimized and traditional process

	Concentration NH_4OH:H_2O_2:H_2O	Time, min	Temperature, °C
Optimized	1:1:0	5	70
Traditional	1:1:5	15	70

6. CONCLUSION

The optimized wet activation process was desirable due to its extensive application in silicon direct bonding. An orthogonal experiments array was designed to investigate the effects of the key parameters which were volume ratio, holding time and treat temperature. For the sake of evaluating surface roughness more reasonably, bear ratio was used as evaluation index instead of *RMS*. The experimental results were analyzed using intuitive analysis method and the effects of process parameters were revealed. Among the three tested parameters, the concentration of activation solution had the most intimate relationship with the surface roughness and the excessively high concentration led to the deterioration of surface roughness. The holding time had obvious effect on the roughness though it was not as strong as the effect of the concentration. As to treat temperature, no remarkable effect was found. Based on the conclusion of experimental analysis, the optimized process was designed and corresponding bonding experiment was performed. The satisfying result proved the validity of the conclusions originated from the orthogonal experiment.

Acknowledgments

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