Effect of dielectric barrier discharge on ceramic surface properties

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Abstract. Ceramics, as a commonly used insulating and thermally conductive material, has a wide range of applications in the field of micro-nano manufacturing. In recent years, research on ceramic bonding has also increased. However, ceramic bonding has some problems such as bonding failure and low bonding quality. Learning from other commonly used bonding materials, the important factor for the success of bonding is the surface properties of the material. Therefore, in this study, the surface of ceramic was treated by dielectric barrier discharge (DBD) to improve surface properties. The effects of dielectric barrier discharge on the surface properties of ceramic is investigated from the three aspects: hydrophilic angle, surface energy and surface morphology. In the dielectric barrier discharge experiment, blind via glass is used as the blocking medium for the upper electrode. The lower electrode is covered by ceramic, and the ceramic is also the material to be treated. The influence of processing time, discharge voltage and discharge frequency on the treatment effect is investigated in the experiment. The hydrophilic angle and surface energy of the treated ceramics are measured and calculated by contact angle measuring instrument, and the surface morphology was observed by scanning electron microscopy. The results show that the longer the discharge treatment time is, the larger the discharge output voltage is, the smaller the discharge gap is, the faster the hydrophilicity of the surface of the ceramic sheet is, and the higher the activation energy is. The damage of the ceramic surface after treatment is small, and the impurities on the surface are destroyed. In general, the surface properties of ceramics after dielectric barrier discharge treatment are better than before.

1. Introduction
Wafer bonding is a cost-effective method for MEMS packaging [1], and it has increasingly become a key technology for material integration in various fields such as MEMS, microelectronics and optoelectronics. Different wafer bonding methods are currently used in the MEMS industry: melting, bonding, eutectic, anode, soldering, etc. [2]. The fusion process requires high temperature annealing and is not suitable for devices with aluminum or copper integrated circuits; bonding bonding is not
highly sealed. At present, the development of low temperature bonding (the highest process temperature is lower than 300 °C) has received more and more attention, it can not only reduce the process cost and time, but also minimize the stress caused by bonding after cooling. And warping. In addition, it is also possible to bond wafers having a large difference in thermal expansion coefficient (CTE). Anode bonding is one of the most common wafer level packaging procedures. In anodic bonding, the substrate is heated to a temperature above 400 °C, a voltage higher than 600 V is applied to the pair of wafers to be bonded, and the migration of electrostatic forces and ions causes chemical bonds to be generated at the boundary layer.

Directly bonded metals and ceramics are particularly useful in high power and high frequency applications where eutectic metal-ceramic bonding is characterized by the presence of an easily identifiable intermediate layer at the metal-ceramic interface. The absence of an intermediate layer will provide good electrical and thermal conductivity at the metal-ceramic junction. Good electrical conductivity in this region accommodates high frequency environments, while good thermal conductivity allows the use of ceramics to directly dissipate heat and improve thermal conductivity.

In plasma processing techniques, it has been well established that plasma generated by exposure to inert gases and/or reactive gases can clean the surface of the material and change its properties, especially its surface energy. The active material from the plasma bombardment and/or reacts with a monolayer on the surface of the material and temporarily or permanently changes its surface properties. [5] Since the adhesion of the group acts on the molecular scale, the plasma technique can effectively achieve the modification of the near-surface region without affecting the overall properties of the material. [6] Dielectric barrier discharge (DBD) has its own advantages. For example, DBD produces more uniform and more active particles than corona discharge, and is less prone to surface damage than the former. In addition, unbalanced, ie "cold" plasma conditions can be established in a reliable manner in atmospheric gas, resulting in significant cost savings.

In this study, a dielectric barrier discharge was used in the atmosphere to produce a non-equilibrium plasma to treat the surface of the ceramic sheet. In the test, the DBD discharge gap was changed to obtain different silicon wafer samples with different treatment effects, and the hydrophilic angle was measured. Scanning electron microscopy (SEM) was used to observe and characterize the sample to investigate the effect on the surface properties of the ceramic sheet.

2. Materials and Methods

This section should be divided by subheadings. Materials and Methods should be described with sufficient details to allow others to replicate and build on published results. Please note that publication of your manuscript implicates that you must make all materials, data, and protocols associated with the publication available to readers. Please disclose at the submission stage any restrictions on the availability of materials or information. New methods and protocols should be described in detail while well-established methods can be briefly described and appropriately cited.

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2.1. Experiment device

The dielectric barrier discharge is placed on one side of the barrier medium to facilitate the dissipation of heat generated during the discharge process. The medium placed on both sides is favorable for generating high-purity plasma [7]. This test is a dual medium, the upper electrode is glass, and the power is off. The grade is alumina ceramic, CTP 2000 K (Coronalab, China) plasma power supply is used to provide high applied voltage. The operating frequency of the power supply can be adjusted from 10 kHz to 40 kHz. The input voltage of the power supply can be adjusted by the voltage regulator. The discharge load consists of an upper electrode, a blocking medium, an air gap and a lower electrode. The schematic of the experimental system is as follows:
2.2. *Experiment materials*
In this experiment, an alumina ceramic sheet was selected as the material to be treated, and the size was 10 mm*10 mm*0.5 mm.

2.3. *hydrophilic angle Experiment*
The free energy of the ceramic surface is calculated by contact angle measurement. The contact angle was measured by the sessile drop technique. 1 μl of the test drop was dropped on the surface of the sample by a syringe. Contact angle measurements were made at 25 °C using a ZJ-6900 contact angle instrument.

2.4. *Effect of dielectric barrier discharge on surface properties of ceramics*
The output voltage of the dielectric barrier discharge device is set to be constant at 7 kHz. Take a piece of qualified test piece to measure its hydrophilic angle, adjust the transformer so that the gap of the dielectric barrier discharge device is 0.2mm, the discharge voltage is 24kV, and the test piece is subjected to dielectric barrier discharge for 90s. The hydrophilic angle is measured immediately after the treatment is completed as shown in Fig. 1. After the dielectric barrier discharge treatment, the hydrophilic angle of the ceramic surface drops rapidly. The reason may be that under the action of the dielectric barrier discharge, the chemical bonds on the ceramic surface are largely broken, and a large number of polar hydrophilic groups are generated, resulting in a ceramic surface exhibiting a hydrophilic state. The surface hydrophilic angle drops from 86.23° to 10.48° as shown.

2.5. *Factors affecting the hydrophilicity of ceramic surfaces*
The influence of these factors on the material was analyzed by changing the parameters using the above test apparatus. In this paper, the parameters such as discharge voltage, discharge frequency and discharge time are selected to study the effect on the hydrophilic angle of the material surface. This test uses a single factor analysis method to specifically analyze the impact of these factors.

First, it is necessary to determine the initial discharge voltage at each frequency. After preliminary tests, it is found that when the voltage is greater than 23 kV, a discharge can be generated at a discharge frequency of 5 kHz to 9.5 kHz. When the peak-to-peak value of the output voltage of the
A high-voltage high-frequency power source is greater than the tolerance value of the air gap, the air gap is broken, and a visible discharge phenomenon between the two metal electrodes is generated, showing a distinct blue-violet discharge beam. As the power supply excitation frequency increases, the output power increases and the discharge intensity increases. At the same time, the degree of unevenness of the air gap electric field increases, and the dielectric barrier discharge is more likely to occur, causing the initial discharge voltage to decrease. The external conditions of the discharge are kept the same, and the dielectric barrier discharge system is energized with a power supply frequency of 5 to 9.5 kHz, and the power supply voltage is between the corresponding initial discharge voltage and the breakdown voltage. Observe the discharge phenomenon of ceramic sheets under different conditions, and record the change of hydrophilic angle with time.

3. Results and Discussion

3.1. Discharge time
It is proposed to select five processing times of 10s, 20s, 30s, 40s and 120s, set the discharge voltage to be constant at 24kV, set the discharge frequency to be unchanged at 7kHz, and before the discharge treatment, the surface of the ceramic sheet has a hydrophilic angle of 113.6°, showing hydrophilicity. Sex. After 10 s of treatment, the hydrophilic angle decreased to 90°, and the decrease was more pronounced as the treatment time prolonged. When the treatment time is 180 s, the hydrophilic angle has been reduced to 14.6°.

![Figure 3. Change in discharge time](image)

3.2. Discharge voltage
Set the power supply output voltage to a constant 7 kHz. The discharge conditions at five different voltages, namely 21, 21.4, 21.8, 23, 25, and 28 kV, were observed, and the change in the hydrophilic angle of the surface was recorded, as shown in Fig. 5. When the discharge frequency is constant, when the voltage is gradually increased from 21 kV to 28 kV, the regularity of the descending speed of the hydrophilic angle is gradually accelerated. At the same time, the rate of decrease of the hydrophilic angle is instantaneously increased at 21.4 kV. After the voltage is continuously increased, the rate of decrease of the hydrophilic angle is not obvious. When the voltage is 21.4 kV and 21.8 kV, the curve of the hydrophilic angle decreases exponentially. When the discharge voltage is greater than 23kV, the hydrophilic angle is less than 15°, and the curvature of the curve is greatly reduced, which is almost closer to a straight line.
3.3. discharge gap
Set the power supply output voltage to 7 kHz and the discharge voltage to 23 kV constant. The discharge conditions at 6 different discharge gaps, namely 2, 2.2, 2.4, 2.6, 2.8, and 3 mm gaps were observed, and the change of the hydrophilic angle on the surface of the ceramic sheet was recorded, as shown in Fig. 5. When the discharge voltage and frequency are constant, when the voltage is gradually increased from 2 mm to 3 mm, the hydrophilic angle is increased from 15.82° to 83.04°. The hydrophilic angle changes slowly when the discharge gap is 2.0-2.4 mm, and the change is accelerated when the gap is 2.4 mm to 2.8 mm. When the gap is larger than 2.8 mm, the hydrophilic angle rapidly increases.

3.4. Surface topography
The surface morphology test was carried out by Karl Zeiss scanning electron microscopy. There were two ceramic samples, one of which was untreated, the other was a constant discharge voltage of 24 kV, and the discharge frequency was 7 kHz. After the gold spray treatment, the image under the scanning electron microscope is as follows, wherein Figures 6a, 6c, 6e, and 6g are the surface of the untreated ceramic sheet, and Figures 6b, 6d, 6f, and 6h are the surface topography of the treated sample.
Figure 6. Surface topography

It can be seen from Fig. 6a and Fig. 6b that the two ceramic sheets have a small surface difference. Further magnification, as can be seen in Figures 6c and 6d, the difference in pore size of the ceramic sheets before and after treatment is still small, indicating that the DBD treatment has little damage to the ceramic surface. Finally, the particles on the surface of the ceramic sheet were enlarged, and it was found that there were large particles on the surface of the untreated ceramic sheet, and the surface particles of the treated ceramic sheet were small, and there were many large particles on the untreated ceramic sheet. The surface particles after the treatment are mostly around 30 microns.
3.5. Discussion

The test relies on the change of the hydrophilic angle of the ceramic sheet during the dielectric barrier discharge treatment and the surface energy to characterize the activation effect of the dielectric barrier discharge, so as to analyze the discharge parameters of the dielectric barrier according to the change curve of the hydrophilic angle to eliminate the hydrophilicity of the ceramic. Influences. The higher the discharge voltage across the electrodes, the faster the hydrophilic angle decreases with time. This is because if the discharge voltage is increased, the output power of the discharge is increased, and more active plasma is generated under the same conditions to destroy the organic functional group, thereby causing the disappearance of hydrophilicity to be accelerated, and the external appearance is observed by the contact angle measuring instrument. The lower surface hydrophilic angle decreases faster. The increase in processing time, in the case of a constant discharge power, brings more reaction time. The reduction of the discharge gap is advantageous for reducing the initial voltage of the discharge and the uniformity of the discharge, so that a better treatment effect can be achieved. The image measurement by scanning electron microscopy shows that the damage caused by the DBD treatment on the surface of the ceramic sheet is small, and at the same time, large particles on the surface of the ceramic sheet can be bombarded into small particles.

4. Conclusions

The test results show that the longer the range, the longer the dielectric barrier discharge treatment time, the larger the discharge output voltage, the smaller the discharge gap, the faster the hydrophilicity of the ceramic sheet surface decreases, and the higher the activation energy. Test results on the surface, the dielectric barrier discharge has little damage to the surface of the ceramic sheet, and can bombard the particles into small particles, and the surface can be closer when bonding, which may be beneficial to the occurrence of bonding.

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