Effect of Oxygen Plasma Treatment on Anodic Bonding

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(Received 12 April 2000)

Oxygen plasma surface treatment of silicon and glass was studied for improving the characteristics of anodic bonding. By using the sessile drop method, we confirmed that the surfaces activated by the oxygen plasma were rendered hydrophilic even at low r.f. power or short plasma exposure time. With increasing plasma power and exposure time the surface roughness was observed to increase. The oxygen plasma treatment was significantly efficient in reducing the impurities on the surface, which caused degradation in the bonding strength and the electrical property in interface. In the tensile test, the oxygen plasma treatment led to a higher bonding strength than the conventional anodic bonding method.

bonding.

I. INTRODUCTION

The anodic bonding of silicon and glass has become one of the important steps in the fabrication of microsensors, fluid handling devices, silicon-on-insulator (SOI) type structures, and many other devices [1,2]. Anodic bonding is an attractive method because it is easy and reliable to control and requires no expensive installation. However, anodic bonding in the environment of a high temperature or a high electric field can bring the degradation of device, so the efforts to reduce temperature and electric field have been conducted [3,4]. In the bonding technology, the state of the surface is important for good quality and reliable bonding, and one method to treat the surface is to utilize a plasma, which has the advantages of simplicity, increased effectiveness, environmental safety, and little effect on other devices during treatment compared with chemical methods using strong acids [5]. The surfaces in contact with the plasma experience interactions which may result in a variety of changes in the surface regions involved in the bonding process. Plasma treatment is thought to an activated and clean surface which results in a strong bond without a high electric field or a high temperature.

In this work, we applied an oxygen plasma treatment to anodic bonding and studied the effect of the oxygen plasma on the surfaces of silicon and glass. Anodic bonding using the oxygem plasma treatment could be II. EXPERIMENTS Corning #7740 (Pyrex) $20 \times 20 \times 0.5 \text{ mm}^3$ glass

performed at lower temperatures and electric fields in the bonding process than it could in conventional anodic

slides and p-type, (100)-oriented $15 \times 15 \times 0.5 \text{ mm}^3$ silicon wafers were used in these experiments. For oxygen plasma treatment, the samples were inserted into a lowpressure radio-frequency (R.F.) plasma chamber. The plasma treatment was performed at a working pressure of 20 mTorr, an oxygen flow of 40 sccm and room temperature. The oxygen plasma power and exposure time were varied from 10 to 150 W and from 10 to 600 s, respectively. Immediately after plasma treatment, the samples were taken from the plasma chamber, put together face to face, and then pressed to form initial contact at room temperature. This assembly was heated on a hot plate, upon which a ceramic insulator had been placed. A glasssilicon assembly was placed between the electrodes, and a direct current (d.c.) voltage was applied across the assembly such that the glass was negative with respect to the silicon.

The surfaces of the oxygen plasma-treated samples were characterized by contact angle measurement, atomic force microscopy (AFM), and Auger electron spectrometry (AES). The contact angle of a water droplet was measured by using the sessile drop technique

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Fig. 1. Contact angle of silicon and glass prepared at various R.F. powers and exposure times.

to estimate the degree of activation of the silicon and the glass surfaces. The more hydrophilic the surface, the smaller the contact angle was. The error range was within \pm 10 % due to the inaccuracy of the angle measurement and the variation in the surface quality, so at least 10 measurements were performed for each specimen. AFM was used to determine the surface roughness of the silicon and the glass substrates. The root-meansquare (RMS) value was obtained by averaging 3 different areas. The cleaning effect of plasma treatment on the silicon and the glass surfaces was investigated by using AES. The bonding strength of the bonded specimens was measured using a tensile strength machine and averaging the results from 3 samples.

III. RESULTS AND DISCUSSION

Figure 1 shows the effect of the R.F. power and the plasma exposure time on the water contact angle for both silicon and glass. Generally, high surface energy solids are hydrophilic and tend to promote wetting, so the contact angle method is an relatively simple and in-



Fig. 2. Surface roughness of silicon and glass as functions of the R.F. power and the exposure time.



Fig. 3. AES survey spectra of wafers treated at various R.F. powers and exposure times: (a) silicon and (b) glass.

direct method for evaluating hydrophilicity [6,7]. At a given time of 60 sec, the contact angle of silicon and glass decreases with power and gradually approaches a saturated value. The lowest contact angle is obtained for the oxygen plasma treatment at a plasma power of 30 W. When the plasma power is fixed at a constant power of 70 W, the plasma exposure time determines the contact angle of the silicon wafer, as well as the glass wafer. With increasing exposure time, the contact angle decreases rapidly during a short-time treatment and then remains at a saturated level. Even at short exposure time and low R.F. power, the surfaces were activated enough to cause the contact angle to be below 10° , which meant that the effectiveness of the oxygen plasma treatment for the surface activation was due to substituting chemically active dangling bonds and hydroxyl groups for the carbon contamination in the surfaces.

The surface roughness is one of the most important parameters in anodic bonding. One of the significant effects of the rough surface is to decrease the bonding strength or even to cause failure of the bonding. The roughness increased with R.F. power and plasma exposure time, as shown in Fig. 2. The oxygen radicals preferentially remove weak Si-Si bonds and break Si-O bonds at the surface. Because the surface roughness plays an important role in the anodic bonding process, high R.F. power or long plasma exposure time, which increase the surface roughness, may degrade the bonding strength.

After oxygen plasma treatment, the carbon impurity



Fig. 4. Relative bonding strength as functions of the R.F. power and the exposure time.

was detected to be below a few at. %, which was lower than in case of SC-1(Standard Cleaning 1) cleaned samples, as displayed in Fig. 3. In the spectrum of silicon and glass, we can recognize the C peak at 272 eV and the O peak at 503 eV. The signal between 60 and 100 eV is composed of Si peaks due to Si-Si and Si-O bonds. In oxygen plasma treatment, the activated oxygen reacts with surface impurities and reaction products were desorbed in the form of volatile materials as follows:

$$C_x H_y O_z + (O_2^*, O) \to CO_2 + H_2 O$$
,

where $C_x H_y O_z$ represents a general hydrocarbon molecule and (O_2^*, O) is chemically activated oxygen: either the vibrationally excited molecule or atomic oxygen. These oxygen radicals preferentially break Si-O bonds at the top of the SiO₂ surface, which enhances the generation of chemically active dangling bonds and hydroxyl groups at the surface of SiO₂ layer. This is in agreement with the results of the contact angle measurement that the oxygen-plasma-activated surface was hydrophilic. This result shows that oxygen plasma cleaning is effective in the removal of impurities, like hydrocarbons, which cause the bonding strength and the electrical property in interface to be degraded.

Activated wafers were contacted and pressed, which caused initial bonding without any thermal treatment or electric field, which made the handing of the bonded wafers easier. The initial bonding strength was about 0.2 ~ 0.3 Mpa, which was not sufficient for an anodic bonding pair. Anodic bonding was carried out when voltages of 250 V were applied at a temperature of 240 °C. The bonding strength as a function of the R.F. power and the plasma exposure time is shown in Fig. 4. The oxygen-plasma-treated specimen had a much higher bonding strength (1.4 ~ 3.3 MPa) than the untreated specimen under the same conditions. However, samples that were overly exposed or subjected to a much higher plasma power showed lower bonding strengths due to rapid increases in the surface roughness during those treatments.

IV. CONCLUSIONS

The effect of oxygen plasma on silicon and glass was investigated for anodic bonding. The influences of process parameters, such as R.F. power and plasma exposure time, on the degree of activation, surface cleanliness, and roughness were investigated by using the contact angle method, AFM, and AES. The oxygen plasma treatment was revealed to be an effective method for the anodic bonding at low electric fields and temperatures and for improved bonding properties.

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