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# CHARACTERIZATION OF BONDED INTERFACE BY HF ETCHING METHOD

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## ABSTRACT

Directly bonded interfaces have been studied using a HF etching method. Bonded wafer pairs were dipped into 50% hydrofluoric acid (HF) solution for 10 minutes and subsequently the etched distance was measured from cross-sectional samples by scanning electron microscopy (SEM). Studied wafer pairs were silicon/oxide, oxide/oxide, silicon/glass, oxide/glass and silicon/quartz wafer pairs. In silicon/oxide and oxide/oxide bonds a relationship was found between the surface energy measured with the crack opening method and the etched distance. In silicon/glass and oxide/glass bonding we found the etch rate of the bonded interface to decrease with increasing bond annealing temperature.

## INTRODUCTION

SOI (silicon-on-insulator) wafers have replaced the conventional silicon wafers in many electrical and sensor applications. SOI wafers are available with different buried oxide (BOX) and SOI-layer thicknesses. The strength of the bonded interface is usually measured with the crack opening method [1]. However, the use of the crack opening method is rather difficult in cases when one or both of the bonded materials are fragile such as glass or thin wafers, since in these cases the blade insertion usually leads to wafer breakage instead of crack formation. Another limitation is that the blade needs to have a place for insertion, e.g. opening between two rounded wafers. Therefore, the surface energy is measurable only from the areas near the wafer edges. The crack opening method has also problems in measuring strong bonded interfaces [2]. In the sensor applications, where structures are released by etching of buried oxide layer, it is important to have a well-defined etch rate and good knowledge about formed oxide wall profile. The objective of this work was to find a new method for bond strength determination especially suitable for fragile substrates.

## EXPERIMENTAL

In the experiments we used <100> oriented 100 mm p-type Czochralski grown silicon wafers, 100 mm Corning 1737F glass wafers and 100 mm fused quartz wafers. Thermal wet oxide was grown on some of the silicon wafers. Some of the wafers were activated in a reactive ion etcher (Electrotech RIE) using argon or oxygen plasma. After plasma activation the wafers were cleaned in 70°C SC-1 ($NH_3$:$H_2O_2$:$H_2O$) solution and/or in deionized water. Wafers were bonded in a commercially available wafer bonder (Electronic Visions EV801). The bonded wafer pairs were annealed for 2 hours at 100°C. After this wafers were cut into 1 cm thick stripes with a dicing saw. The stripes were annealed at various temperatures to achieve bonds with different bond strengths. For comparison, some of the stripes were used to measure the surface energy with the crack opening method [1]. The samples were dipped in a 50% HF solution for 10 minutes. Subsequently, the samples were cleaned in deionized water and dried. The etched distance was measured from the cross-sectional samples by using scanning electron microscopy (SEM).

RESULTS

A logarithmic relation between the surface energy and the etched distance was found for silicon/oxide and oxide/oxide bonds (Fig. 1). For strongest bonds, the etched distance was ~15 µm and the etched oxide profile was almost straight (Fig. 2), showing that the etch rate was nearly uniform across the oxide. For weak bonds, the oxide profile shows that etch rate is higher at the bonded interface than on the other parts of the buried oxide (Fig. 3).

We also performed the HF etch experiments for plasma-activated silicon/oxide bonds. The etch rate versus measured surface energy curve shown in Figure 4 differs significantly from the one obtained for silicon to oxide bonds formed without plasma activation. The etched distance in a plasma activated sample annealed at 200°C was from 80 µm to 100 µm depending on the activation procedure and the measured surface energy was ~2400 mJ/m$^2$. Etched distance in silicon/oxide wafer pair bonded without plasma activation having similar bond strength was about ~15 µm. Higher etch rate in the plasma activated samples may be due to porous or damaged layer formed into the oxide surface during plasma activation. The layer affected by the plasma ions is found to be about 5nm thick and it is quickly consumed during SC-1 cleaning [3].

In thermal oxide to glass bonding we measured the length of the notch (Fig. 5) formed during etching. The length decreased with increasing annealing temperature (Fig.6). Due to the fragility of the glass wafer, the applicability of the crack opening method was limited to weak bonds. The measured surface energy for oxygen plasma activated oxide to glass bonding annealed at 200°C for 2 hours was ~680 mJ/m$^2$.

We investigated the suitability of HF etching method also in silicon to quartz bonding. To avoid debonding due to the mismatch in thermal expansion coefficients [4], we used oxygen plasma followed with SC-1 cleaning to achieve strong bonding at the annealing temperature of 200°C. The crack opening method was again not applicable for bond strength measurement, because of the fragility of the quartz wafer. The etched distance was ~126 µm (Fig.7), which is close to the etched distances measured for similarly prepared silicon/oxide wafer pairs (~100µm). Since the etch rate for quartz glass and thermal oxide in HF are comparable, this result suggests strong bonding between quartz and silicon.

CONCLUSIONS

Etching in hydrofluoric acid is a promising method to measure the bond strength in cases where the crack opening method cannot be used. This method enables bond quality measurements from bonded fragile wafers, ready-made SOI and diced chips. The HF etching method can also be used to create bond strength maps over the wafer area. Limitations for the method are that at least one of the materials at the interface must be attackable by the HF and the etch rate is not only dependent on the surface energy, but also on the sacrificial material properties and possible interfacial voids.

We have shown a relation between the etch rate and the surface energy in conventional silicon to oxide and oxide to oxide bonding. By using plasma activation, high surface energies can be obtained for silicon/oxide bonds at annealing temperatures below 200°C. However, the etch rate of the buried oxide for the plasma activated wafer pairs is much higher than for non-activated silicon/oxide bonds having similar surface energy values. We believe that this difference in the etch rates is due to the porous or damaged layer at the plasma activated surface.

The method was also studied for silicon to glass, oxide to glass and silicon to quartz bonds. In silicon to glass and oxide to glass bonding, the HF formed a notch near the bonded interface. The length of the notch was found decrease with increasing annealing temperature.

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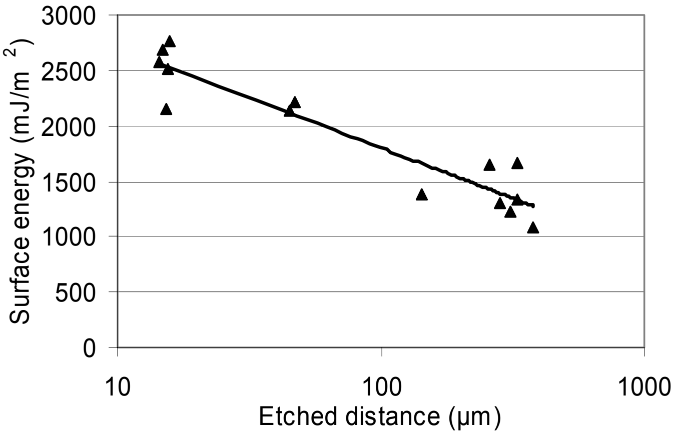


**Figure 1.** The measured surface energy as a function of the measured etched distance for the bonded SOI structures. The etching was carried out for 10 min in 50% HF at room temperature. No plasma activation was used.

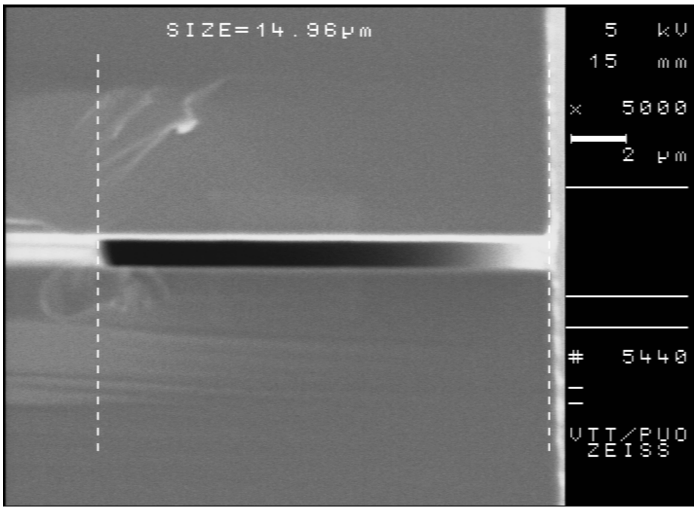


**Figure 2.** SEM image of bonded silicon to 1µm thermal oxide wafer pair. Annealing was done at 1100°C. The etched distance is ~15 µm. Surface energy was found to be ~2600mJ/m$^2$.

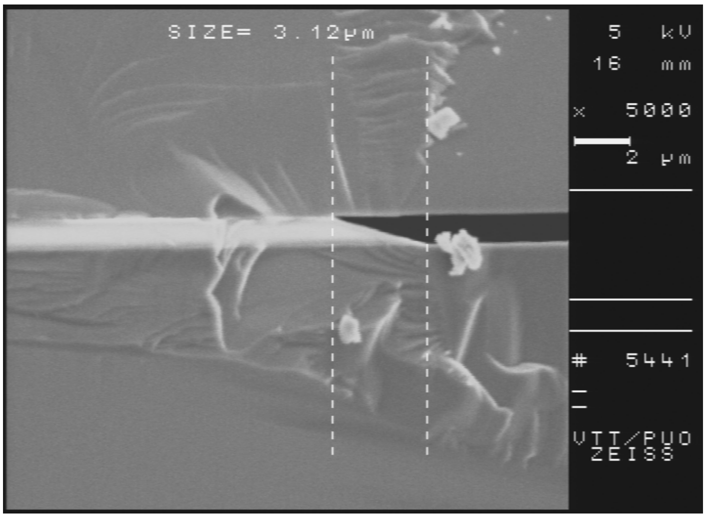


**Figure 3.** Profile of the etched oxide wall on 50nm oxide (top) to 1000nm oxide (bottom) bond annealed at 1150°C. Total etched distance was ~50µm and surface energy was ~2200mJ/m$^2$.

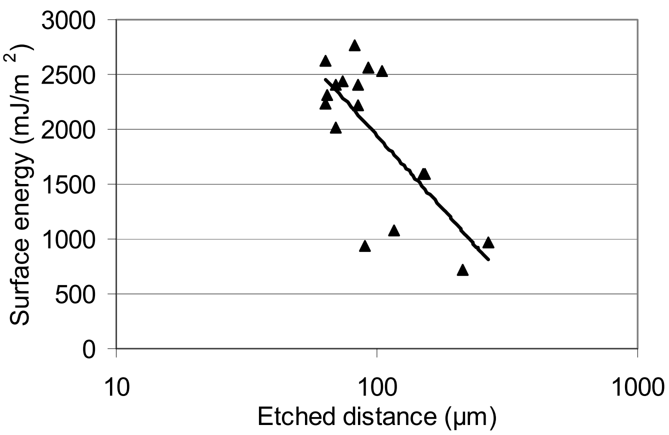


**Figure 4.** The measured surface energy as a function of the measured etched distance for plasma-activated and bonded SOI structures. The etching was done for 10 min in 50% HF.

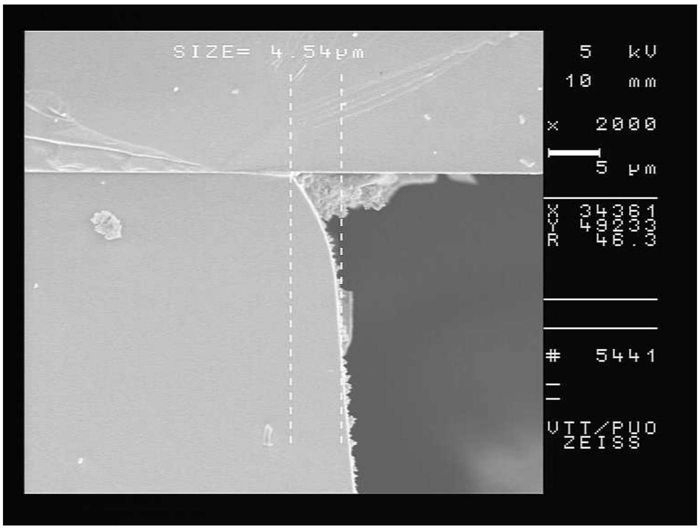


**Figure 5.** SEM image of the notch in the plasma activated 100nm thermal oxide (top) to glass (bottom) bond. The bond annealing temperature was 500°C. The notch was created by etching the sample for 10 min in 50% HF. The length of the notch is ~4.5 µm.

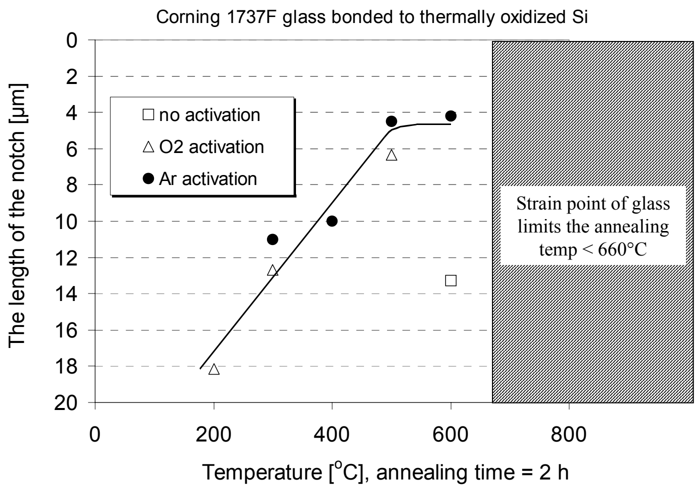


**Figure 6.** Measured length of the notch as a function of annealing temperature for 100nm thermal oxide to glass bonding with and without plasma activation.

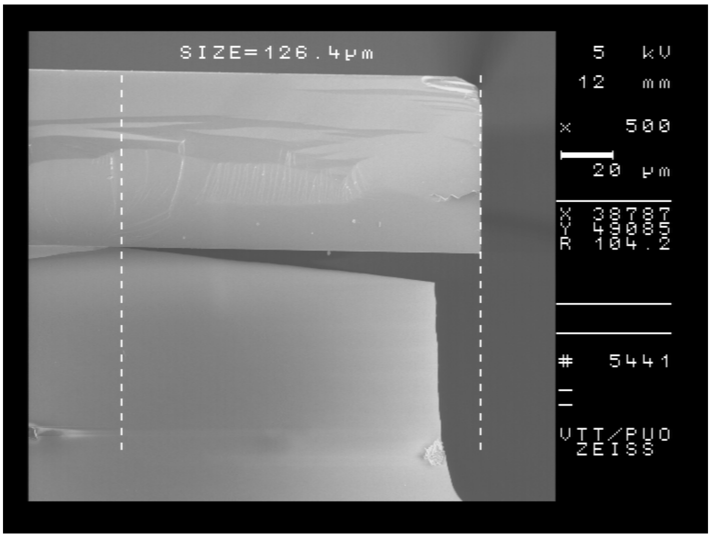


**Figure 7.** SEM image of bonded silicon (top) / quartz (bottom) wafer pair. Wafers were activated in oxygen plasma followed with SC-1 cleaning. Bond annealing was carried out at 200°C. The etched distance is ~125 μm.