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How to cite:

Masteika, V.; Kowal, J.; Braithwaite, N St.J and Rogers, T. (2013). The effect of atmospheric moisture on crack propagation in the interface between directly bonded silicon wafers. *Microsystem Technologies*, 19(5) pp. 705–712.

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Version: Accepted Manuscript

Link(s) to article on publisher's website:

<http://dx.doi.org/doi:10.1007/s00542-012-1685-8>

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The effect of atmospheric moisture on crack propagation of the interface between directly bonded silicon wafers

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Abstract

Infra-red video sequences were taken of directly bonded silicon wafer pairs undergoing the razor blade crack length test for bond strength in a specially designed jig. A series of tests were carried out under controlled atmospheres of nitrogen at various relative humidities. Analysis of the video images showed that the crack continues to propagate rapidly for several minutes after the blade has stopped moving, and that the presence of moisture has a strong positive influence on the rate of crack propagation under static loading. A new Maszara protocol is suggested based on modelling crack growth using our experimentally derived constants.

1. Introduction

The ability to bond silicon wafers to each other without the use of intermediate layers is important in the manufacture of MEMS as it avoids the risk of introducing thermal stresses in service. A bonding process that entails heating the wafers to a temperature above 800°C has been in use for some years and produces very strong bonds with high reliability [1]. This process is useful for the manufacture of substrates for certain microelectronic applications, but is less useful for MEMS, where ideally the bonding would be carried out as one of the final steps. This calls for much lower process temperatures to avoid adverse effects on other materials that may be present on the wafers, such as aluminium, gold, or doped regions. Suitable procedures have been invented, typically involving a surface activation step, such as brief exposure before bonding to an oxygen plasma [2]. These processes enable strong bonds to be formed without the need to raise the wafer temperature above 2-300°C.

These lower temperature methods result in bond strengths that are less than those that are routinely obtained with the high temperature process, and moreover there is greater variability in the bond strength. Low temperature direct silicon bonding is therefore being actively researched. The successful development of a bonding process that is robust and repeatable enough for industrial use depends on the reliable measurement of bond strength. Many approaches have been tried, but the razor blade crack length test devised by Maszara [1] (Figure 1.), also known as the Dual Cantilever Bending test (DCB), has become a widely used method.

Figure 1. Maszara Test

The Maszara formula (1) links wafer thickness w , crack length l , half the wedge thickness u and the Effective Youngs Moduals of the beam E^* in order to give the bond energy γ in J m^{-2} .

$$\gamma = \frac{3E^* u^2 w^3}{32l^4} \quad (1)$$

The test consists of inserting a razor blade into the interface between the wafers and measuring the distance between the tip of the crack thus formed and the edge of the blade. This is done by taking an infra-red shadowgraph of the bonded wafer following insertion of the blade and taking measurements from the image.

The DCB test tends to produce a spread of results, even for different parts of the same wafer pair. Part of the reason for this is the dependence of the estimated bond strength on the measured crack length raised to the fourth power (1), which makes the result very sensitive to small errors in the measurement. These can arise from the quality of the imaging (alignment, focusing, and image resolution). Another important source of error is variations in the elapsed time between insertion of the blade and the capture of the image, as it has been observed that the crack continues to propagate after the blade has stopped moving[3, 4].

In these low temperature bonded wafer pairs, the bond is formed between the native oxide surface layers on the silicon (or, in some cases, between the thicker oxidized layers formed on silicon wafers by thermal oxidation at an early stage in the process sequence). The crack can therefore be regarded as propagating in a material that is chemically similar to silica. Studies of the mechanisms of crack propagation in silicate glasses [5-7] suggest that this occurs by different processes on a number of different time scales, the shortest of which is very rapid – of the order of milliseconds.

The purpose of the work reported here was to discover whether crack propagation phenomena similar to those found in glass could be observed in the interface between two silicon wafers, and hence whether or not they need to be accounted for in bond strength measurements. In addition the role of atmospheric moisture, proposed as active in the weakening of bonds at the crack tip in glass, was investigated. If this was found to be important for silicon direct bonding, then it too would have a bearing on how crack length tests should be conducted.

2. Stress Corrosion

Stress corrosion in silica glasses is a well-characterised field [7]. Stress corrosion in silica occurs when a sub critically strained siloxane bond is broken by interaction with a water molecule, causing it to revert back to separate silanol groups as illustrated in Figure 2. and equation (2).

Figure 2. Stress Corrosion Reaction

The siloxane bond between two bonded silicon wafers can be thought of as a type of silica glass, a much-studied material. Before the glass cracking literature can be compared to Maszara testing it is important to appreciate the change in relationship between crack length, crack front speed and stress introduced by the differing standard sample arrangements illustrated in Figure 3.

Figure 3. Sample setup for dual cantilever bending

- (i) **Typical materials science setup, this leads to an increase of stress at the crack tip as the crack length increases**
- (ii) **Maszara setup, the crack tip stress decreases as the crack length increases**

A constant force arrangement is common when cracking materials as illustrated in (i). As the crack grows this increases the displacement of the two beams and hence the leverage on the crack tip. Therefore stress on the crack tip will increase with time. During the Maszara test the initial displacement is fixed and so stress on the crack tip will decrease with time as the crack grows.

Crack Growth Regimes

In the stress corrosion literature [7] it is generally accepted that there are five regimes of crack growth in silica.

Figure 4. A standard logarithmic graph of crack growth rate against stress, adapted from [7]

$K = \text{Stress at crack tip}$

$v = \text{Crack velocity}$

The stress at the crack tip K is related to the bond strength γ extracted from crack measurements using the Maszara formula by formula (3) and the crack velocity v is the velocity of the crack front.

$$K = \sqrt{2E^* \gamma} \quad (3)$$

Region O: At and below K_I bond breaking is equalled by bond creation and the crack is stationary.

Region I: Between K_I and K_{II} the crack is growing principally due to stress corrosion limited by the available stress.

Region II: Between K_{II} and K_{III} the crack is still growing due to stress corrosion but growth is limited by the available moisture. This region is defined by the rate at which moisture is able to diffuse to the crack tip, which is a function of the relative ambient humidity

Region III: Between K_{III} and K_c the crack is growing due to thermal fluctuations in subcritically strained bonds.

Region IV: At and above K_c , the stress is equal or greater to the bond strength and so the crack grows sonically.

Regimes O to III are a function of both the availability of water to drive the corrosive reaction and the stress at the crack tip. In region O there is not enough strain for chemical corrosion to outpace bond reformation so any crack remains stationary. In region I there is ample time for atmospheric moisture to diffuse to the crack tip and the rate of crack growth is determined purely by the available stress. In region II the crack is moving so rapidly and using up the available moisture so quickly that the reaction rate is limited by the transport of moisture to the crack tip. As an environment becomes more humid it is expected that the gradient and length of this region will decrease. In region III stress corrosion is no longer occurring, however the crack is still growing due to thermal fluctuations. In the absence of moisture this

gradient would extend down to very low crack velocities, as indicated on the sketch graph.

During Maszara testing we begin with a $K > K_c$ and so start in region IV. As the crack lengthens with time, K decreases and the crack growth profile moves back down through the regions. Eventually we end at region O when no further crack growth occurs.

3. Experiment

Substrate preparation and description of apparatus

Bonded wafer pairs were produced using the oxygen radical activation technique [8]. These were annealed for 1 hour at 200°C after bonding. Crack propagation measurements were made on these wafer pairs using the apparatus shown in Figure 5.

Figure 5. Apparatus for controlled crack propagation by razor blade insertion

1. Motor
2. Lead Screw
3. Mobile jig
4. Tensioned Shim
5. Bonded Si Wafer Sample
6. Sample Clamp

DCB testing was performed using the above debonding jig. A 125 μm thick hardened steel shim, held under tension, was used as the wedge. The square leading edge of this shim allowed the length of the crack to be accurately measured from the infra-red transmission images. Images and video were taken using a modified Microsoft LiveHD webcam with the IR blocking filter removed and custom optics installed. This included an IR slot filter with a pass band from 0.8 to 2 μm , which lies both within the transmission window of the silicon wafers and the range of wavelengths detectable by the webcam's imaging array. The VirtualDuB software program was used to record uncompressed videos and extract frames.

The DCB testing jig was placed inside a glove box to provide a controlled humidity environment as shown in Figure 6.

Figure 6. Glovebox for providing a controlled environment

1. Microsoft LiveHD webcam
2. CCD Optics
3. IR pass filter
4. Picoscope RH-O2 Humidity Sensor
5. IR Lamp
6. Desiccator
7. Bonded wafer pair
8. De-bonding jig

For dry operation the chamber was first evacuated to 0.8 atmospheres in order to aid in the removal of lab air and then flushed for 2 hours with N_2 , passed through the desiccator. The flow was then cut off and desiccated silica gel exposed in the chamber. DCB testing then commenced once humidity had stabilised. After testing the silica gel desiccant was removed.

For high humidity experiments ceramic containers of hot water were placed inside the chamber and the humidity allowed to stabilise before DCB testing commenced.

Humidity was continuously measured using a Picoscope RH-O2. The humidity sensor was calibrated using a small volume sealed glass container. To establish zero humidity the container was partly filled with desiccated SiO_2 and the humidity probe inserted. To establish 100% humidity the container was cleaned and then partly filled with boiling water and the humidity probe inserted.

Experimental procedure

A modified DCB testing procedure was used in order to minimise uncertainties and maximise the number of cracks that could be recorded. Each wafer was first diced into four equal width strips with a diamond edged saw as shown in Figure 7.

Figure 7. Wafer dicing pattern

A standard razor blade was used to open a pre-crack in the end of each sample. This pre-crack was not recorded and was purely created to ease the insertion of the square-edged jig shim. To produce a DCB measurement the cracked sample was then loaded into the debonding jig and the shim inserted into the pre crack. Setting the camera to record a video the shim assembly was then further inserted. Our previous work has indicated that initial crack growth occurs at a maximum rate of $15\text{-}20\text{ mm s}^{-1}$. The shim was therefore rapidly inserted at a rate greater than 20 mm s^{-1} for a distance sufficient to advance the crack, and then made to stop within the time between two consecutive video frames. The resulting crack was then recorded for approximately 5 minutes. This procedure was repeated until the entire sample was debonded.

Three bonded wafer pairs were created under identical conditions and diced as described above. DCB testing was performed under three humidity conditions, 5% 34% and 90%, with one sample from each wafer being entirely debonded at each humidity. The remaining samples were used as spares where fracture through the bulk silicon caused a tested sample to fail prematurely.

3. Results and Discussion

Stress Corrosion Curves

Measurements made from the recorded videos produced a large data set of crack lengths as a function of time, humidity, wafer thickness and initial bond strength, which we know varies across our bonded wafers. This data was smoothed and processed using the Maszara formula in order to produce normalised stress corrosion curves that were then analysed further. A selection of this processed data is presented in Figure 8.

Figure 8. Normalised Stress Corrosion Curves at three humidities

Comparison of crack length time series at three different humidities suggests a systematic dependence on humidity and so a simple mathematical model was proposed for $L = f(\phi, t)$. This is given in equation (4) and illustrated in figure 10.

$$L = L_o(1 - L_i e^{-\frac{t}{\tau}}) \quad (4)$$

L = Crack length/ mm
 L_o = Final crack length/ mm
 L_i = Ratio between final and initial crack length
 t = time/ s
 $\tau = f(\phi, T)/ s$
 ϕ = relative humidity/ %RHA
 T = Temperature/ K

Figure 9. Example of model fitted data for $\tau = 19$ s, $\phi = 5.3$ %

Although this model begins to deviate from the data for to long term and initial growth, it was successfully fitted to all available curves and values of $\tau = f(\phi)$ were extracted. These were then plotted and fitted as shown in Figure 10.

Figure 10. Model τ fitted against experimental data

These results strongly suggest a systematic dependence of crack growth rate in DCB bonded wafers on humidity. This agrees well with previous results and theory. Physically we explain τ as being a time constant that is inversely related to the probability of a water molecule reacting with a strained but unbroken siloxane bond and so this data indicates a non-linearity in that relationship.

Velocity against stress

Whilst our previous work sheds some light on to the role of humidity it does little for our ability to fundamentally understand crack growth or predict bond strengths.

In Figure 11. the crack front velocity at various humidities is plotted on a log-linear scale against apparent bond strength γ , in J m^{-2} .

Figure 11. Crack front velocity for wafer RHO3 against apparent bond strength, at various humidities

It is apparent that our crack growth matched that typical of stress corrosion literature as sketched in Figure 4. Therefore we were now able to employ that body of work in analysing our data. As described in [7] and [6] there are a number of equations that are commonly used to represent region I, II and III. There are two broad approaches to describing crack growth data: energy balance, and the chemical activity of the crack front, both of which yield analogous results. We chose to use the chemical

activity family of models originally proposed by Wiederhorn et al [9] as we are specifically accounting for chemical corrosion in our experimentation.

We elected to model regions I and III using separate equation, (4) and (5) neglecting region II.

$$v_{vac} = v_0 \exp \left[\frac{-(\Delta E_a - (b.k))}{R.T} \right] \quad (5)$$

$$v_{stress} = v_0 \exp \left[\frac{-(\Delta E_a - (b.k) - p)}{R.T} \right] \quad (6)$$

Where:

$$\begin{aligned} v_{vac} &= \text{crack growth rate in a vacuum} / m s^{-1} \\ v_{stress} &= \text{humidity-enhanced crack growth rate} / m s^{-1} \\ v_0 &= \text{velocity at } k_c = 15 ms^{-1} \{a\} \\ b &= \text{area molar density of Si-O-Si bridges} / m^2 mol^{-1} \\ k &= \text{crack tip stress} / J m^{-3} \\ R &= \text{molar gas constant} \\ T &= \text{temperature} / K \\ \Delta E_a &= \text{activation energy of 1 mol of Si-O-Si} = 139 \text{ kJ mol}^{-1} [\text{ref}] \\ p &= \text{activation energy moisture reduction factor} / J mol^{-1} \\ \{a\} &= \text{Our own experimental data} \end{aligned}$$

Equation (4) is taken directly from Wiederhorn et al. [9] and describes crack growth in the absence of any chemical process. It represents thermally-activated breaking of bonds at the crack tip. The initial bond strength is encoded into variable b , the area molar density of Si – O – Si bonds. The strength of the bond between two wafers is a function of the density of bridging Si – O – Si bonds. Previous work has measured the number density of the precursor Si-OH bonds [10] but this approach allows the post anneal bond number density to be extracted.

Equation (5) describes the stress corrosion region and is a departure from the commonly used Wiederhorn equation for region III equation (6).

$$v_{stress} = v_0 a_0 \left[\frac{P_{H2O}}{P_{atm}} \right]^\eta \exp \left[\frac{-(\Delta E_a - (b_2 k))}{R.T} \right] \quad (7)$$

Where:

$$\begin{aligned} a_0 &= \text{Humidity constant} \\ P_{H2O} &= \text{Partial pressure of water} \\ P_{atm} &= \text{Atmospheric pressure} \\ \eta &= \text{A constant close to 1} \\ b_2 &= \text{Humidity exponent constant} \end{aligned}$$

In this approach the effect of moisture is expressed in terms of available surface absorption sites and approximated by a langmuir isotherm [6] outside of the

exponential. Our approach moves the humidity constant into the exponent to highlight its role as an enthalpy barrier reducer p .

Our data was then smoothed using Mathcad's spline fitting smoothing on the apparent bond strength vs velocity curves and then fitted with equations (4) and (5). An example of the fitted data is shown in Figure 12.

Figure 12. Model fitted smoothed experimental crack growth data

All of the v vs γ curves were then fitted and the values of p plotted against the ratio of the partial pressure of water vapour gas (P_{H_2O}) to total atmospheric pressure (P_{atm}), a literature convention we adopted [ref].

Figure 13. The dependence of the activation energy reduction parameter p on the partial pressure of water vapour.

The experimental data was then fitted with a power law (8).

$$p = r \left(\frac{P_{H_2O}}{P_{atm}} \right)^q \quad (8)$$

Where:

$$r = 122611 \text{ J mol}^{-1}$$

$$q = 0.1047$$

Proposed new Maszara test mode

Using the above two equation approach it is theoretically possible to determine the initial bond strength using only a single velocity measurement from region I, the relative ambient humidity and the temperature of the wafer. In order to test this method we performed DCB tests on a 2nd batch of wafer samples under similar conditions to those used to define (6). Figure 16 is a comparison of the bond strengths as obtained from rapid initial crack length measurements with those derived from the extrapolation of region I crack length data using the analytical model.

[Figure 16, new wafer series being created. Will show initial bond strengths by mobile Maszara vs modelled values]

Figure 16 relative disparity between measured and modelled bond strength as a function of humidity

As can be seen the model is able to predict crack lengths with an x% uncertainty. This reduces the need to obtain very rapid initial crack lengths and to open significant new crack for each measurement, and also presents an alternative to driving the wedge at a speed v_0 , which may be difficult with fragile or strongly bonded samples and requires a debonding jig.

We conclude that the most accurate bond strength measurements are obtained by driving the wedge at v_0 . The high-speed video capture of newly-opened cracks provides the second most accurate results, with the stress corrosion technique introducing further uncertainty. However all three of these methods yield reproducible results and eliminate the problem of crack growth that plagues this otherwise simple and spatially sensitive technique.

4. Conclusion

We have shown that stress corrosion in DCB testing is the same as stress corrosion described in silica. We have also proposed a model of crack propagation speed based on fundamental physical principles that is a function of relative humidity, temperature and initial bond strength. Finally we have suggested a new DCB protocol utilising our model that enables the recovery of initial bond strengths from crack growth affected measurements.

Acknowledgments

We would like to thank the EPSRC and AML for their support and partnership in this work undertaken as part of a CASE funded studentship.

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