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Plastic Deformation of Thin Si Membranes in Si-Si Direct Bonding

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The effect of bond anneal in Si-Si direct bonding of laminates with thin membranes suspending closed cavities is studied. For membranes of a certain size and thickness, it is found that the under-pressure in the cavity during bond anneal leads to plastic deformation of the membrane. By controlling the cavity pressure it is found that the Si crystal of the membrane can be kept intact during bond anneal.

Introduction

High temperature annealed silicon direct - or fusion - bonding is a widely used process, enabling the fabrication of robust, long-term stable devices with 3D structures [1]. To achieve good bond strength a temperature above at least 900 °C is required [2]. For device fabrication a wider thermal range is required, with oxidation and diffusion processes up to 1150 °C. During these high-temperature steps, thin membranes suspending sealed cavities may become permanently deformed. We have investigated the extent and cause of such deformations and we have established how these deformations can be avoided by adjusting the cavity pressure in the bonded device prior to the high temperature anneal step.

Experimental

Fusion bonding employs pre-bonding in controlled atmosphere at typically 50 °C. The bond anneal furnace works at atmospheric pressure, and a desired maximum temperature of 1050 °C was used in this study. Assuming that the gas inside the closed cavity follows the ideal gas law, a pressure of 247 mbar at 50 °C corresponds to 1 atm at 1050 °C. In this study, pre-bonding pressures of 207 – 247 mbar N_2 were chosen to achieve a sufficiently small deflection of the membranes at the 1050 °C anneal step. In addition, one wafer pair was pre-bonded in vacuum as a reference.

Test structures were fabricated on eight 100 mm wafer pairs. SOI wafers with 43 µm device layer, 500 nm Buried Oxide layer (BOX) and 380 µm handle wafer were used, together with 400 µm thick bulk wafers. Both wafer types were p-type with (100) orientation. The bulk wafers were oxidized, and the oxide was patterned to allow for etching of an inlet hole after bond anneal.

Membranes were fabricated by Deep Reactive Ion Etching (DRIE) of the SOI wafers through the handle wafer, using the BOX as an etch stop. Circular membranes with a diameter range of 840 – 3720 µm and square membranes with side edges 1105 – 3270 µm were defined, as summarized in Table 1. The same lithography mask was used on the front side of the wafers to etch the oxide on top of the membranes, followed by

TABLE I. Summary of test structure dimensions, calculated maximum stress at 1 atm pressure difference, and crystal property results for pre-bonding in vacuum

Cavity name	Membrane type and size	Calculated max stress @ 1 atm [MPa]	Bonding results in vacuum
S1	Square 1105*1105 µm	25	Intact crystal
S2	Square 1885*1885 µm	74	Slip lines
S3	Square 2465*2465 µm	126	Slip lines
S4	Square 3265*3265 µm	221	Slip lines
C1	Circle dia 840 µm	7.2	Intact crystal
C2	Circle dia 1880 µm	36	Slip lines
C3	Circle dia 2640 µm	72	Slip lines
C4	Circle dia 3720 µm	142	Slip lines

thermal oxide growth. This makes a step in the Si on the front side, making it easier to identify the membrane position after bonding. To ensure symmetric membranes all masking oxide and the exposed BOX were stripped, and a new 190 nm thick thermal oxide was grown.

Wafer pairs were cleaned in piranha, rendered hydrophilic in RCA-1, aligned, and loaded into an EVG510 wafer bonder, separated by 50 µm thick spacers. The bond chucks were kept at 50 °C. The chamber was brought to vacuum ($<1*10^{-3}$ mbar), and purged with N_2 to reach the desired pressure level. Wafers were brought to contact, and a bond force of 1000 N was applied for 2 min. A cross section of the test structures is shown in Figure 1. A Zygo New View white light interferometer (WLI) was used to measure membrane deflection. For each of the pre-bonded laminates, some reference membranes were measured before the laminates were annealed at 1050°C for 2 hours. After bond anneal, wafers were measured again, and an inlet to each cavity was etched by DRIE. A final deflection measurement was then performed. The membranes were also inspected by an optical microscope with a halogen lamp and a Hamamatsu C8800 IR sensitive camera.

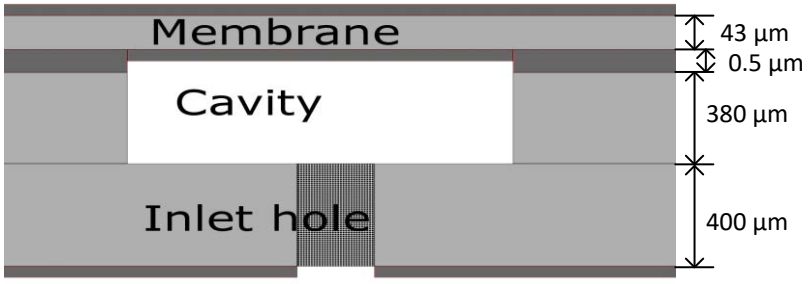

Figure 1. Sketch of test structure cross-section (not to scale). The inlet hole is etched by DRIE after bond anneal.

The maximum membrane stress was calculated at 1 atm differential pressure, using the standard analytical expressions [3] for flat plates in the linear regime. At the relatively small resulting deflections, this approach is sufficiently accurate, as evidenced by comparison with the FEA derived values by Ren et al. [4]. The corresponding calculated maximum deflections range from 0.04 µm to 13.5 µm for the circular membranes, and 0.15 – 11.4 µm for the square ones.

Results

The WLI measurements before bond anneal of the S4 type membranes showed deflections of around 12 µm for the pressure compensated membranes, and around 15µm for the membranes of the vacuum reference wafer, in good correspondence with calculated values.

After bond anneal, WLI measurements of the pressure compensated membranes showed essentially the same deflection as before for most devices, with exception for the lowest part of the compensating pressure range combined with the largest membranes. The vacuum cavities, however, showed severe increase in deflection. Profile plot from WLI measurements of a vacuum cavity C3 type membrane after bond anneal is shown in Figure 2, together with fringe pattern picture from the WLI microscope. The C3 and S3 membranes had a deflection of around 5 µm before bond anneal, which changed to 50-56 µm after bond anneal. The C4 and S4 vacuum membranes bend in a way that gave problems with range limitations in the WLI measurements, but a deflection of 108 µm was estimated on one S4 membrane.

Inspection of the membranes in an IR capable microscope revealed a clear difference between the vacuum and the pressure compensated membranes. Figure 3 shows IR images of two S4 membranes after bond annealing: one pre-bonded in vacuum, and one pre-bonded in a 207 mbar N_2 ambient. What we interpret as slip lines were clearly seen in all the vacuum pre-bonded square and circular membranes except the smallest of each type, as concluded in Table 1.

An IR microscope image of a C2 type membrane pre-bonded in vacuum is shown in Figure 4. Slip lines are visible all over the membrane, and not only in the regions that are expected to see the highest stress.

Ventilation of the membranes by opening the inlet holes provided little change in deflection of the vacuum pre-bonded reference cavities. The pressure compensated

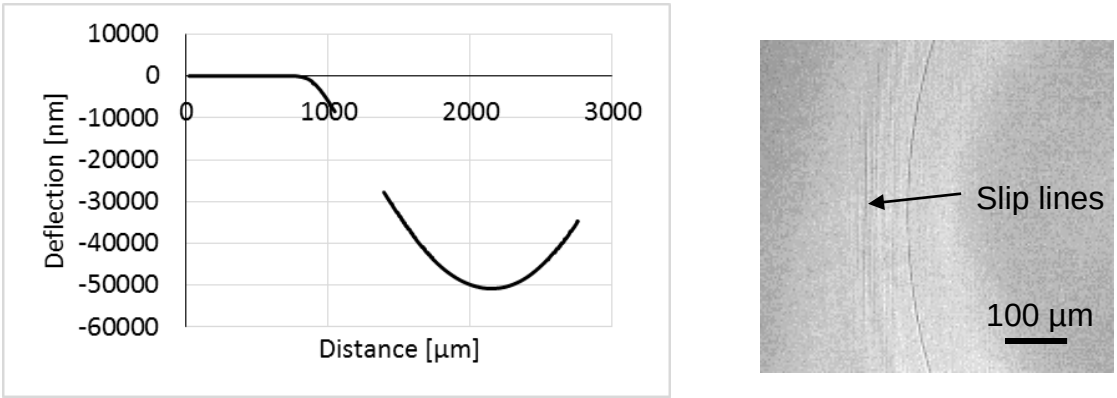

Figure 2. WLI measurement of a C3 type membrane pre-bonded in vacuum, after bond anneal (left). The break in the otherwise solid line appears because the wide angle of reflected light cannot be picked up by the WLI microscope. The right-hand picture shows the fringe mode picture from the WLI, with visible slip lines. Bar in lower right corner indicates 100 µm length.

membranes however essentially turned flat (+/-0.1 µm), except for many C4 and S4 membranes pre-bonded at 207 mbar. Deflection measurements for these two membrane types vs pre-bonding pressure are shown in Table II. Figure 5 shows WLI measurements of different C4 membranes before and after INLET etch. For membranes from 207 mbar cavities, both types of "after INLET"-deflections shown in Figure 5 were seen, and deflections less than 100nm are excluded from the 207 mbar averages of Table II. In Figure 5, please note that the visible surface step does not coincide with the actual membrane edge. This step was only intended as a crude means of making the location of the membrane visible from the front-side; thus, the two involved lithography masks were not precisely aligned. Furthermore, the DRIE that defines the membrane produces a negative etch angle and a slightly larger membrane. The deflection of the non-deformed membranes is not symmetric relative to this step, but it is not clear if this is due to misalignment alone or to the asymmetric stress imposed by the misaligned step. The most important information from this figure lies in the maximum deflection values.

TABLE II. Average deflection and maximum calculated stress at bond anneal conditions for C4 and S4 after INLET etch vs pre-bonding pressure, for pressure compensated membranes. 1 atm pressure is assumed on the outside. Deflections less than 0.1 µm are excluded from average for 207 mbar pre-bond pressure, as these devices fell into two different categories; one with and one without permanent deflection.

Pre-bond pressure [mbar]	**Average deflection, C4 [µm]**	**Average deflection, S4 [µm]**	**Max stress @1050 °C, C4 [MPa]**	**Max stress @1050 °C, S4 [MPa]**
207	1.42	1.28	23	36
232	0.08	0.09	9	14
247	0.08	0.09	0	0

By visual inspection before bond anneal it was noted that the membranes of pressure compensated cavities fell into two categories on each laminate: "large" and "small"

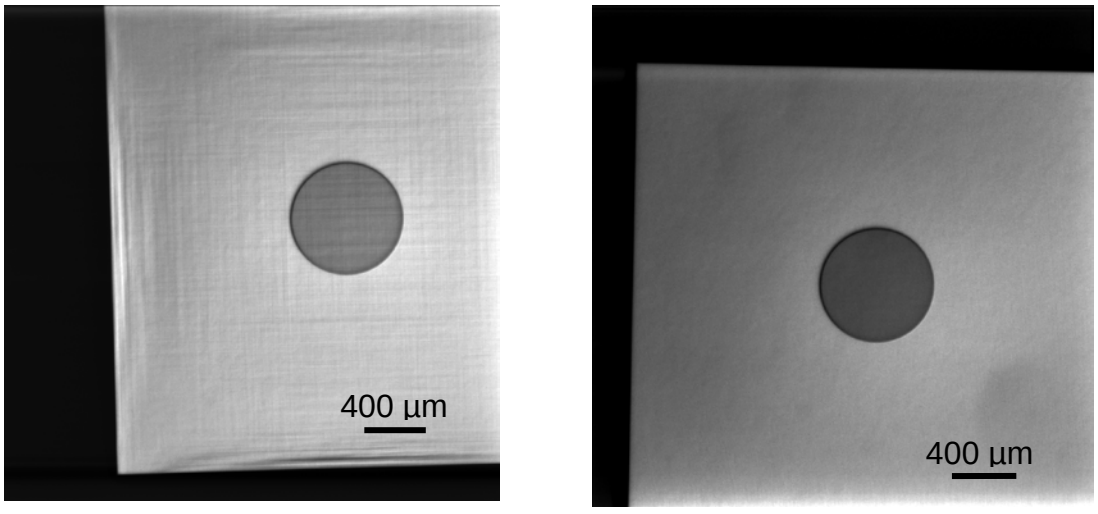

Figure 3. IR microscope picture of S4-type membranes pre-bonded in vacuum (left) and at 207 mbar (right). The light gray area is the membrane. The straight lines seen in the left hand membrane are slip-lines in the Si crystal. The dark circle is the oxide mask opening for inlet DRIE. Bar indicates 400 µm length.

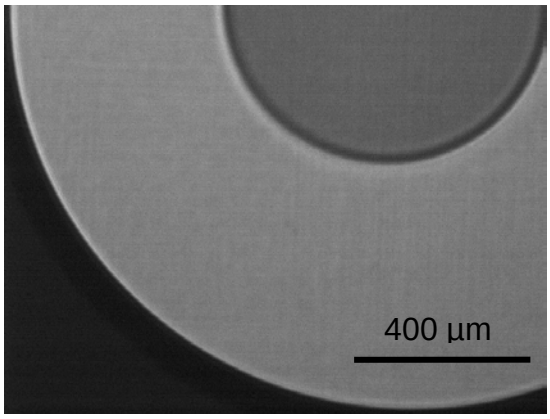

Figure 4. IR microscope picture of parts of a C2 membrane pre-bonded in vacuum. It is worth noting that there are slip-lines all over the membrane. Bar indicates 400 µm length.

deflection. The wafer positions of some of the membranes with "large" deflection before bond anneal were noted, to check if membranes at these specific locations behaved differently in bond anneal. It was found that after bond anneal these membranes also had slip lines and deflections of the same level as the corresponding membranes on the vacuum reference wafer. An example of this is shown in Figure 6, where the permanently deflecting membranes are easily distinguishable.

Discussion

The maximum stress for a 1 atm pressure difference over the membranes is calculated by analytical expressions assuming deflections in the linear regime, as shown in Table 1. Our calculations show that a 1 atm pressure difference over the S1 and C1 membranes corresponds to a stress in the membranes of 25 MPa and 7.2 MPa, respectively. Both

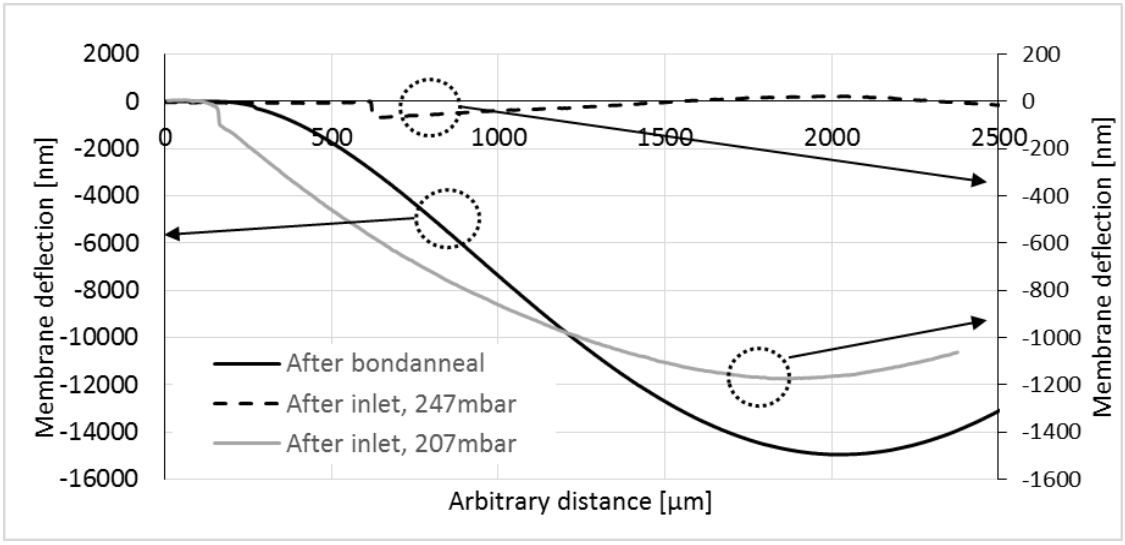

Figure 5. Profile plots from WLI measurements of C4-type membranes just after bond anneal and after INLET etch for 207 and 247 mbar. Please note the difference in scale. After INLET etch the membrane location has been made visible by the step created in the Si before bonding, but note that this step does not coincide precisely with the membrane edge.

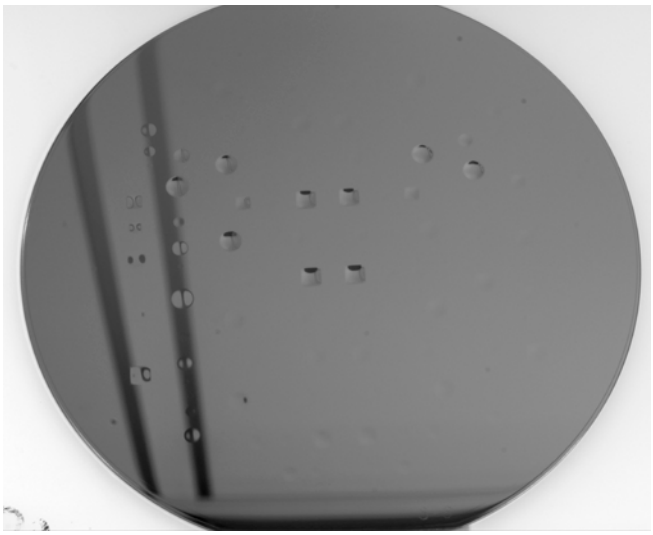

Figure 6. Picture of a 100 mm diameter wafer pre-bonded at 207mbar, after bond anneal. The membranes with visually clear deflection were found to show slip-lines similar to those seen on the vacuum reference wafer.

these smaller membrane types were found to have intact Si crystal after pre-bonding in vacuum. That C1 behaves this way corresponds well with the results in the recent work by Ren et al. [4], who report on similar experiments accompanied by FEA simulations. The fact that S1 with 25 MPa stress seems to be below the yield strength warrants further discussion, see below. All the other - hence larger - membranes were found to have slip lines when pre-bonded in vacuum, a result that also corresponds well with Ren et al. [4], as our calculations show a membrane stress of ≥36 MPa for these geometries.

Ren et al. used a Si yield strength value at 1000 °C of around 20 MPa [4]. They referred to Patel et al.'s experimental work [5], from which they extracted yield strength values at various temperatures; 700, 800, 900, and 1000 °C. For this extraction process, they chose a specific strain rate from Patel's three rates. Since the strain rate is a critical parameter in discussions regarding the dynamic process of silicon yield, we include some clarification and discussion on this theme here. The experimental temperature dependence of the Si yield strength was summarized in Figure 14 in Patel's work, for the case of initially dislocation-free Si crystals. (The data are for the maximum stress σ_M of the stress-strain curve, which is also referred to as the "upper yield point".) The experiments were performed by pulling a 2.5 cm long silicon specimen in a fixture, with constant pull rate (and thus, constant strain rate) at varying temperatures. Patel et al. provide these data at three different "crosshead velocities", i.e. pull rates. These rates must be divided by the 2.5 cm total length in order to obtain the strain rate. Thus, to the best of our understanding, ref. [4] chose Patel's data for the lowest strain rate out of the three, $3.3*10^{-5}$ s^{-1}. This is not an obvious choice *per se*, but the corresponding maximum stress values are indeed those in [5] that best fit Ren's experimental results.

In our work a temperature of 1050 °C is used. If we extrapolate the above-mentioned curve from [5] to 1050 °C, we obtain roughly 12 MPa. This is clearly lower than the calculated 25 MPa that our S1 devices experienced without noticeable plastic deformation (although in accord with the C1 situation). The 25 MPa value is in better agreement with Patel's data for their *medium* strain rate, as extrapolation of the medium strain rate curve to 1050 °C gives a maximum stress of about 27 MPa. This strain rate of

$3.3*10^{-4}$ s^{-1} is ten times larger than the rate used in [4]. This difference demonstrates that caution is required when establishing design and process guidelines for membrane yield. Improved understanding of involved effects, of correlations between modelling and experiments, and of correlations between different experimental situations, is needed.

The large dependence of yield behavior on strain rate was further investigated by Sumino [6], as well as the influence of initial dislocation density and of oxygen contents. Sumino as well as Patel furthermore stress the fact that yield is a highly dynamic process, and Sumino carefully discusses the generation, motion, and multiplication of dislocations due to strain. It may be fruitful to try to reason around the question of how strain rates and other parameters in such experiments as Patel's and Sumino's are relevant in our experimental situation. Such experiments as theirs are characterized by *steadily increasing strain* on a bulk sample under tension with *no bending*, at a *constant temperature*. In contrast, our situation is one of i) a near constant strain (except during a very limited period of gas expansion in the case of gas filled cavities), ii) a strongly bending membrane (which accentuates the role of microscopic details in the silicon surface rather than the bulk situation of the pull experiments), and iii) a temperature that is in fact steadily increased until the final 1050 °C is attained. These are far from two identical cases. The most obvious difference between the two experimental situations is the increasing strain as opposed to the constant strain, but the other factors can certainly not be disregarded.

In [4] it is reported that the larger membranes continue to evolve toward somewhat larger deflection between 1 hr and 16 hrs at the anneal temperature. (In fact, they also report negative creep in some cases.) In our work, we have used an anneal time of 2 hrs at 1050 °C, a time that falls between Ren's values. This should be noted when comparing results. In addition, we speculate that our anneal temperature ramp protocol may also have implications. This could be a matter of interest for further studies.

Another remarkable observation in [5] and [6] is how much the upper yield stress increases with diminishing initial dislocation density. Although modern silicon wafers typically are delivered with densities that are much lower than those of the highest range presented in these works, this is another reason for caution when comparing different works. In fact, Sumino presents data that do not go below $2*10^4$ cm^{-2}, which may still be a somewhat higher density than in modern silicon wafers such as those used in our study. Arguably, most silicon wafers today can probably be assumed dislocation-free in this regard.

For those of our wafers that were pre-bonded with a cavity pressure of 247 mbar, the estimated membrane stress is close to zero at bond anneal conditions. All membrane types that were pre-bonded between 233 and 247 mbar were found to be slip-line free in IR microscope pictures, and they turned essentially flat after INLET etch. This proves that this accuracy in the compensating pressure range is sufficient to avoid permanently distorted membranes, even for 43 µm thick membranes as large as at least 3265*3265 µm square.

Wafers pre-bonded at 207 mbar cavity pressure also had flat membranes except for some of the C4 and S4 types. 207 mbar at pre-bonding corresponds to 847 mbar at bond anneal, which gives a calculated maximum stress of 23 MPa and 36 MPa, respectively,

for the C4 and S4 membranes. As can be seen from Figure 5, even the C4 types can show some permanent distortion, but no slip-lines are visible in IR microscope. (Future studies could incorporate pit etching to better reveal whether dislocations exist.) As the wafers pre-bonded at 207 mbar show a combination of undistorted and distorted C4 and S4 membranes, the stresses of these membranes are probably close to the onset of plastic deformation for the bond anneal process used in this study. Uneven gas filling cavity-to-cavity and/or pre-bending of the wafers might give just the amount of stress variation needed for plastic deformation in some wafer positions and no deformation in others.

The highest stress occurs close to the membrane edge. Hence, one would expect that in membranes that are just above the border for plastic deformation, slip-lines would be observed only close to the membrane edge, while for membranes far above this border slip lines could be expected for the entire membrane. The latter is indeed the case for e.g., S4 in Figure 3 (left), pre-bonded in vacuum. For the C2 membranes pre-bonded in vacuum, that with their calculated stress of 36 MPa arguably are around the border values, the slip lines also appear all over the membrane, as in Figure 4. On S4 and C4 membranes pre-bonded in 207 mbar, slip lines are not visible at all, despite the remaining deflection after INLET etch, as seen in Figure 5. The calculated maximum stresses for "C2 in vacuum" and "S4 at 207 mbar" are nearly equal (both around 36 MPa), and they both experience plastic deformation. Yet, the slip-line observations give very different results. It seems that crystal dislocations are too small to be revealed by IR microscopy in the C4 and S4 membranes pre-bonded at 207 mbar, but they are clearly visible on C2 membranes pre-bonded in vacuum. These observations are also candidates for future studies.

When the wafers have been loaded into the bond chamber they are kept apart with 50 µm thick spacers only. Thus, the clamping force from the bond chuck, possibly combined with wafer bow, can easily make the two wafers touch each other in some areas. During the pump stage of the pre-bonding, remaining gas in the cavities will have a chance to escape even where wafers touch; but during the chamber filling stage the under-pressure of the cavities will suck the two wafers together, thus hindering gas filling of cavities. This is most likely the case for the wafer pictured in Figure 6. Thicker spacers and slower gas filling were in a later study found to increase the gas filling yield.

Conclusion

In conclusion, our work shows that flexible silicon structures such as membranes can be permanently deformed during bond anneal if a stress exceeding the yield strength of silicon is present during anneal. This deformation can be avoided by applying a compensating N_2 gas pressure during pre-bonding, thus actively reducing the differential pressure across the membranes during bond anneal. Crystal slip lines can quickly be detected by IR-sensitive microscope inspection for severe crystal slip, but can be difficult to identify when the dislocations cover a limited area of the crystal. Our results provide guidelines for proper design. We underline, however, the need for carefulness, as devices and experimental conditions are not necessarily directly comparable across technologies and set-ups, and dislocation generation, motion, and multiplication are complex and dynamic processes that still require further studies in order to be properly understood and quantified in the context of silicon device design and implementation.

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