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This report results from a contract tasking University of Uppsala as follows: The proposed work contains two parts, first the ordinary literature survey work with a summary report including conclusions, and second, some experimental work supporting the design of a high pressure check valve. Two test structures are foreseen to be used. 1) A direct fusion bonded wafer samples with different surface activation prior to bonding shall be exposed to high pressure Hydrogen for a significant period of time. After burst test of all samples shall the bonding strength be calculated and a statistical analysis performed. 2) The second test structure shall be used to examine embrittelment of springs in mono crystalline silicon. The stiffness and strength of very small beams shall be measured and analyzed. Two reports will be delivered, a midterm report halfway through the study, and a final data package including a summary report at end of contract. A conference contribution is foreseen to be a part of the final data package.				
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HYDROGEN EFFECTS ON SILICON MICROSYSTEMS

Final Report

February 15, 2009

Ref: Grant no. 063055

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1 STUDY BACKGROUND

1.1 General

There is a growing awareness in society that fossil fuels as used today must be replaced with renewable more environmentally friendly alternatives. In many respects, hydrogen gas is the ultimate alternative, it can be created by splitting water into oxygen and hydrogen with many clean renewable energy sources and when used the byproducts are energy and plain water again. The retrievable energy can be used in conventional combustion, powering cars, heating houses, etc. or be used together with fuel cells to produce electricity in large or small scale. The energy content per kilo is very high for Hydrogen, almost three times higher than gasoline. However it is a concern if there are some fundamental problems related to the use of hydrogen. The diffusion rate through most materials is quite high, as the hydrogen molecules are very small, hydrogen embrittlement can be a safety issue, etc The concept in the background for the Study on "Hydrogen effects on Silicon" is a new approach for very effective high pressure storage using many small autonomous tanks each with a silicon micro system controlling the gas flow. It is absolutely fundamental for the concept that there are no unknown effects on mono crystalline silicon by Hydrogen exposure over time.

1.2 Hydrogen MacroSpheres concept

. Hydrogen is stored under high pressure in a large number of small autonomous sub-tanks, the MacroSpheres, in the concept. The difference between internal and external pressure controls the gas flow to or from the MacroSpheres. Advanced Micro System Technology (MST) is the key enabling element in the gas handling chip which is mounted inside each sub-tank. The system combines the best storage performance available with focus on a convenient and non-dramatic handling for the user. Some system highlights are presented in the following. In the concept, each small spherical tank contains its own gas handling system, integrated in form of a chip on the inside of the spherical tank, see figure 1.2-1 below.



Figure 1.2-1 Chip location inside a MacroSphere

In the filling procedure a large number of MacroSpheres are placed in a high pressure filling tank and when the pressure is higher than the pressure inside a MacroSphere a check valve opens a flow path, through a filter structure and a flow restrictor, from the outside to the inside. When the pressure difference decreases down to close to zero, the check valve closes again. The second important device in the outlet flow path is a pressure-controlled valve, which is closed under another given limit, this limit can also be freely chosen, but should typically be a little higher than the normal atmospheric pressure. This means that the refilled Macro Spheres can be transported between the filling station and the consumer in leak-tight condition i.e. without danger and loss of hydrogen, or particular requirements on the transport vehicle, even in an open paper bag.

The fluid system which permits both filling and release of the hydrogen in a controlled way, is a silicon chip with several micro mechanical devices working together in a small stack of silicon wafers. The wafer stack consists of five micro machined wafers, two thicker protection wafers on both sides of three thin wafer with a number of very tiny mechanical structures etched out. In figure 1.2-2 is a cross-section through the stack depicted.



Figure 1.2-2 Wafer stack cross-section

In some valve structures tiny suspension beams are used to support the valve cap, in others are thin membranes used. It is important that this tiny structures keep their mechanical properties also after a long exposure to Hydrogen under high pressure. A typical valve cap, with a diameter of 20-30 micrometers is depicted in figure 1.2-3 below.



Figure 1.2-3 A suspended valve cap.

1.3 Hydrogen effects on Silicon

When using hydrogen gas in combination with micro systems, systems normally are implemented in monocrystalline silicon wafers, some basic questions immediately arise. One of them is the question if there are any unknown unwanted effects in the interaction between Hydrogen gas and monocrystalline Silicon that prevent or complicates the use of Micro System Technology in the handling of Hydrogen gas. This is the objective for this study. Three tasks constitutes the study, to manufacture, test and evaluate a number of test samples with respect to bond strength for one type of test structure, study possible voids growth in the bond interface after a long duration exposure of Hydrogen under high pressure and embrittlement of narrow beams in an other type of test structure.

2 HYDROGEN EXPOSURE PROCEDURE

2.1 Storage Chamber design

The chamber is design is a cylindrical form with a lid at each end as presented in the intrim report, see figure 2.1-1 left. The flanges at the end of the cylindrical body have connections to the gas line and pressure control system. Material is stainless steel.

The inner diameter is set to 45 mm in order to make the chamber usable even for pressurizing test of the macrospheres later on, as the macrospheres have their outer diameter of 40 mm.



Figure 2.1-1: Cut view of the H2-embrittlement test chamber (left) and an exploded view of a sample cassette (right).

The cassette supports two rows of samples very secure, each sample is supported on two sides. The main reason is to prevent that the samples are mixed by accident when removed from the chamber. The system has been working well.

2.2 Hydrogen exposure procedure

The storage chamber was directly connected to a conventional gas cylinder with maximum pressure 180-200 Bar, the pressure was monitored frequently. But even as the system was located outdoors in a locked gas container, we was not permitted to have an open connection between the bottle and the storage chamber. That in turn created a small problem as the volume with high pressure Hydrogen around the samples become pretty small and it was difficult the have the system leak tight enough for a constant pressure over the whole storage period, instead the chamber was refilled frequently and the exposure time extended to compensate for the pressure drop, in order to have the same result as if the samples should have been exposed to 200 Bars for 1,2 and 3 months.

3 BOND STRENGTH TEST SAMPLES

3.1 Sample design

The bond strength test chip consists of two bonded silicon wafers where each has a thickness of $525\mu m$. The bottom wafer contains $50\mu m$ deep cavities where etch cavity consist of a smaller gas trench and a chamber which is pressurized during the burst tests. A picture of the wafer pair is depicted in figure 3.1-1 below. The top wafer sealing of the cavity has a trough etched visa, for feeding in the gas when the cavity is pressurized.



Figure 3.1-1 Design Bond strength test sample

3.2 Sample manufacturing

The total number of manufactured wafers where 6 bonded pair where three included direct fusion Si-Si bond and the other three direct fusion Si-SiO₂ bond. All wafers, independent of the type of bond interface, has been manufactured with exact same parameters in similar steps in order to make good comparative possible in later evaluation phase. All bonded pairs of wafers have been analyzed by IR-camera at wafer level before and after the high temperature bond annealing and oxidizing step (2000 nm at 1050 C) After annealing the chips was ID marked and diced to individual chips. The chips where voids have been detected are documented on chip level. The samples are manufactured in batches on four inch wafers; each bonded wafer pair contains 22 bond test chips. Both the cavities on the bottom wafer and the vias at the top wafer has been processed by a wet isotropic etch step using 40% wt KOH at 70°C.



Figure 3.2-1 Picture of one of the cavity wafers (W9) with KOH etched cavities. The cavity deep is measured to $52\mu m$.



Figure 3.2-2 – Each bonded wafers pair contain 22 chip. This picture shows the identification numbers for each chip seen from the cavity wafer side. I.e. the via opening are on the backside in this figure.

3.3 Annealing effects on voids

Wafer package NR:	1	Bonding I/F:	Si-Si fusion	Remarks
Before annealing		After annealing		All pictures are taken from the cavity wafers side. (See Figure)
	2.4		• • •	There where some problems during bonding these wafers. It took about 5min in the bond-equipment to find the relevant alignment marks. This can have resulted in contamination of the surfaces.
P11_3-06-PP001_B1_1	NR1_1.bmp	P11_3-06-PP00	1_B1_NR1_6.bmp	A large un-bonded area at the alignment chamfer. This seems however to have been much smaller after the annealing step. Affected chips are after annealing C20-22.
				Two small voids remain after the annealing step. Both can also be seen in the figure above.
			• • • •	Affected chips after the annealing seem to be in the boundary between C20 and C21 and a single void at chip C12.
P11_3-06-PP001_B1_1	NR1_4.bmp	P11_3-06-PP00	1_B1_NR1_5.bmp	
		50 m	+	A lot of small voids after the pre- bonding step that also seems to remain after the annealing step.
				Affected chips is C1, C2 and in the boundary between C6 and C7.
ph g	0	• •		NOTE. Not picture has unfortunately been taken of the chips C4 and C5 after the annealing step.
P11_3-06-PP001_B1_1	VR1_2.bmp	P11_3-06-PP00	1_B1_NR1_9.bmp	remaining voids.

3.4 Hydrogen exposure

After marking and dicing where all test chips without disturbing voids randomly selected to four groups to be the reference group and 1,2 or 3months group respectively. All void samples where threated as a 3 months group.

3.5 Bond strength investigation

The original plan was to use the storage period to refurbish the old bond burst equipment, which had been used at the Ångström Space Technology Centre with good results. The first discovery made was unfortunately that the old equipment was so dismounted and scattered that is was impossible to put it together again. It was decided to build a new and more integrated unit, but for many reasons was the design and manufacturing so delayed that it was decided that it was no meaning to test the now old samples, a different type of test has to be done, see chapter 8, Future verification work.

3.6 New Bond Burst equipment

The basic design of the new equipment is briefly presented in this section. Instead for using high pressure direct from a standard gas bottle in the lab which always is a complication it was decided to use the "House gas" (Nitrogen,6 Bar pressure) and a pneumatic pressure amplifier to generate the burst pressure, see figure 3.6-1 below.



Figure 3.6-1, Bond Burst Equipment, Block diagram

A separate feed line for the burst gas gives a possibility to use other gases in the sample interface, see future verification work. The large pneumatic cylinder is pushing a small piston forward until the remaining dead volume become very small and the gas pressure high. The generated pressure is continuously monitored and as soon as a sudden pressure drop is noted is the peak value recorded and the pneumatic cylinder reversed. Two short stroke cylinders also used in the system, one prevents the sample from falling down into a scrap box before bursting and the other is pressing the sample against the test block. The equipment is made user friendly as several safety switches are involved, so a sample can not be bursted if not in position and properly protected by safety lids.



Figure 3.6-2 Bond Burst Equipment, Cross-section

The test block is milled out from solid high strength aluminum with very narrow gas channels, the expected maximum pressure should be well above 100 Bar which is more than enough for most test structures. A more detailed cross-section though the test block is given below.



Figure 3.1-3 Test Block Cross-section

3.7 Void growth investigation

. The void is un-bonded area and will therefore reduce the bonding strength on chip level making it useless for bond strength tests. The sample is however of high interest for analysis a possible void growth in a hydrogen environment, for which samples with natural voids are needed.

Wafer chip	IR-picture before hydrogen storage	IR-picture after hydrogen storage 6 months	Comment
W1 C7	P11_3-06-PP001_B1_W1_C7.bmp		No difference can be seen between the chips.
W1 C8	P11 3-06-PP001 B1 W1 C8.bmp		No difference can be seen between the chips.
W1 C20	P11_3-06- PP001_B1_W1_C20.bmp		No difference can be seen between the chips.
W1 C21	P11_3-06- PP001_B1_W1_C21.bmp		No difference can be seen between the chips.

3.8 Conclusion Voids Growth

The pictures above is only a few of the available, but the result is the same for all. No void growth has been detected due to the Hydrogen exposure. That voids kan change their size is clearly indicated in the pictures before and after the annealing step, where many voids shrinks or disappear during the treatment.

4 STATIC BEAM TEST SAMPLES

The purpose with this test is to examine if there is a detectable embrittlement effect on very thin beams of monocrystalline Silicon when they have been exposed to high pressure Hydrogen under a long period of time. The current plan is to use of a micro manipulator inside a SEM to measure the stiffness and strength of a large number of beams and then make a statistical analysis of a possible effect. Currently is the Micromanipulator being reinstalled in one SEM in the clean room. It has not been is use for some years. If the installation fails for any reason is the back-up plan to use a modified Nanoindenter, now used for a similar purpose at a University in France.

4.1 Design static beam test sample

The static beam are small strait beams in the wafer plane. They have dimensions between 50-100 micrometer and a length of 1-5 mm. The dimensions resembles of the spring used in the micro mechanical hydrogen storage system under development, the MacroSpheres. They are etched out from a single silicon wafer by use of deep reactive Ion Etch. The area around the beams has been thinned down to approximately 100 micro meters with conventional wet etch. The rings at the end of each beam serves the purpose to give a well defined target for the micro manipulator tip when pushing down the beam. All rings are on a strait line in order to simplify the handling, the manipulator has only to move in one axis. The beams have five different lengths in order to give a possibility to eliminate unwanted side effects of the measurement set-up.



Figure 4.1-1 Test chip with 10 beams



Figure 4.1-2 Detailed design test chip.

4.2 Sample manufacturing

The plan is to manufacture a minimum of 300 small beams. The reason for the large number is that it can be difficult to define the dimensions in the root of each beam, which has a high influence on the measured maximum bending strength. Two wafers with 15 structures 14.4x20mm each containing 10 beams is under manufacturing and 2/3 of them are planned to be in the Hydrogen storage within two weeks. The remaining 1/3 is use to create a baseline. Two separate wafers were manufactured, called W7 and W8. After manufacturing, W8 was put in oxidation oven (~2.7 μ m thick silicon oxide) and subsequently stripped of oxide in order to round off sharp edges and corners. W7 was left untreated.

After through-etching W8, the carrier wafer used in the DRIE process did not separate from the wafer as supposed. In some way the thermo-tape adhered to the surfaces, possibly burned in the DRIE process or due to low pressure between the surfaces. This was however solved by putting the wafers in 7up, effectively dissolving the thermo-tape and freeing the wafers from each other without damage.

When through-etching W7, the carrier wafer was attached using small parts of thermo-tape instead of a whole tape, and this time the wafers could be separated easily. However, the wafer looked burned on the backside after the DRIE process.

Figure 3.1 shows W7 with 19 intact chips and one destroyed, probably due to tension from the DRIE step. Some of the beams collapsed during the DRIE step as well. Figure 3.2 shows two intact chips more in detail.



Figure 4.1 A manufactured wafer (W7) with 19 intact chips.



Figure 4.2 Close-up of two intact chips on W7.

There was some residue left on the wafers after the DRIE etching; this could be seen as "burn marks" on many of the beams on both wafers. Figure 5.3 shows two beams from W8 photographed in optical microscope, one with residue and one without.



Figure 4.3 Left: beam (from W8) with residue ("burn marks") from DRIE, Right: beam in the same chip as the left picture with no residue.

4.3 Hydrogen Exposure

The silicon chips are stored in hydrogen atmosphere for various time accordingly to table 1 below.

Chip no.	Chip no. From wafer Storage time in		Comments	
	no.	hydrogen atmosphere		
		0 (ref chip)		
		0 (ref chip)		
C16	W7	7 days		
C7	W8	7 days	Pin 2 missing	
C6	W7	1 month	Pins 7,8,9,10 missing	
C14	W7	1 month	Pin 3,7,8 missing	
C20	W7	1 month		
C3	W8	1 month		
C11	W8	1 month		
C15	W8	1 month		
C4	W7	2 months	Pin 3, 4, 5 missing	
C10	W7	2 months	Pin 2,3,4 missing	
C12	W7	2 months		
C1	W8	2 months		
C9	W8	2 months		
C17	W8	2 months	Pin 5,6,7,8 missing	
C2	W7	3 months	Pin 5,6,7,8 missing	
C8	W7	3 months	Pin 9 missing	
C18	W7	3 months	Pin 6,7,8,9,10 missing	
C5	W8	3 months		
C13	W8	3 months	Pin 2 missing	
C19	W8	3 months	Pin 6 missing	

4.4 Test equipment

The equipment used when breaking the beams is MINIMAN II (further described in document **Test equipment and test plan for H2_Embrittning effect on mono crystalline silicon bulk Silicon**). The silicon samples are placed in a small holder (see figure 4.4-1), and the holder is mounted in MINIMAN II (figure 4.4-2)



Figure 4.4-1. Sample holder with mounted silicon sample.



Figure 4.4-2. MINIMAN II

4.5 Test results

The manufacturing of the silicon structures used in this experiment is described in the document **P11_3-07-DRT-014**. The beams in the silicon structures are very fragile, and are easily break during handling. Due to this, some chips do not have complete amount of pins. Note that the definition below is made with the side of the chip that is marked with the chip id no facing upwards! However, pin 1 and 10 are identical, pin 2 and 9 are identical (and so on). Identical pins are handled as one test group for each chip



Figure 4.5-1. Definition of pin

The silicon chips are stored in hydrogen atmosphere for various times accordingly to table 1 below.

Totally, 18 chips are included in the study (marked yellow in Figure 2). Wafer 7 and wafer 8 have been manufactured mainly in the same way. The difference between them is that wafer 8 has been oxidized. Chips marked with yellow in Figure 2 indicates stored in hydrogen, white chip indicates no storage.



4.5.1 Equipment verification

Ref chips from w7 and 8 were used to test and verify the equipment. Totally four chips were used. Only the shortest pins (pin no. 1 and pin no. 10) on each chip were stiff enough to break on each chip. Due to this, only the results from pins in position 1 and position 10 will be used in the results.

Chip no	Pin 1	Pin 2	Pin 9	Pin 10
Ref 1	Missing	Did not break	Broke	Broke
Ref 2	Broke	Broke	Missing	Missing
Ref 3	Broke	Did not break	Did not break	Broke
Ref 4	Broke	Did not break	Did not break	Missing

4.6 Test data handling

The information logged in the computer connected to the test equipment contains

- Time (clock)
- Normal force in arm

Displacement

A measurement of actual displacement during tests was not possible, and the speed of the needle was unknown. The latter was – however- constant. In order to obtain the actual displacement of the pins, the movement of the bend needle was measured without any chip mounted. For ten times the displacement of 10 seconds was measured.

Average displacement for $10 \text{ s} = 1,5\text{mm} \Rightarrow \text{Average speed} = 0,15\text{mm/s}$

Since time was logged during the measurements, the displacement could be obtained by using the average speed above.

Applied force

The normal force in the arm holding the needle was logged. This corresponds directly to the applied force.

Applied force = Logged normal force

Data used for evaluation

The data files from the measurements contained many measurements. The data used were the last ten (10) measurements before breakage (including the breakage point). All other

measurements were disregarded in the evaluation. The clock was transformed to displacement using the average speed calculated above (i.e 0,15mm/s). Results



Figure 4.6-1. Overview of results.

The measurements show no signs of changes in brittleness between the four groups.

4.7 Discussion

The equipment used for the measurements contained many sources of error and the measurements shows fluctuating curves. However, the overview of the different groups (different storage time) presented in figure 4 show no differences *between* the groups and this result should be regarded as reliable but handled as an indicating result. No numerical values should be regarded as reliable since there was no way of checking the accuracy of the logged normal force. Hence, the numerical accuracy of the measurements is questionable. However, the equipment was used in the same way for each measurement and the potential inaccuracy should be constant, causing no impact on the comparison between the different measurements.

The needle forcing the beams to bend and break was placed in the round markings on the pins during tests. Due to this, the tip of the pin was "stuck" to the needle, causing a slightly forced displacement path. This may have caused further inaccuracies in the logged normal force (since this introduces a force vector with an angle). However, this should not have any impact of the fact that no differences were visible between the different groups.

The test chips used for this experiment were very brittle, and the handling of them before tests caused many pins to break. This reduced the amount of pins to test.

All pins 1 and 10 were breakable, but only some of the pins 2 and 9. When the experiment was formed, the design was set so that all pins should brake in the tests. However, the pins turned out to be much more flexible than intentioned. This caused further reduction of pins to tests.

The computer connected to the test equipment was very old, and only allowed logging of test results (no further handling of the data). All files needed to be moved to another computer by the use of old floppy disks in three steps and much data was lost due to the fact that six files were corrupt and five files contained data but lost names and log heads during the moving of the files. The latter could not be used due to the fact that they contained measurements of unknown chips and pins. All of this reduced the usable test results.

4.8 Conclusion Static Beam test

The conclusion from the rather rough tests performed is certainly that there is no dramatic embrittelment effect on the beams, it should have been noted. It was decided not to extend the test series with the equipment available, but rather use a different test set-up, see future verification work below.

5 FUTURE VERIFICATION WORK

5.1 Static embrittelment time effect

In spite of that no papers has been found indicating an embrittelment effect nor has the static beam test resulted in such an indication it is hard to say that the effect do not exists. Some scientists claims that it could possibly be a time dependent effect, as fast as it occurs when exposed to Hydrogen just as fast will it disappear (by diffusion) when the sample is removed from the hydrogen exposure. In order to disprove this theory is a new test planned in which a thin membrane is exposed to and bursted in the same set-up using the Bond Burst Equipment with a different type of sample.

5.2 Sample manufacturing

The new sample reassembles on the outside about the bond strength test chips but is really different on the inside. It is a three wafer stack with an inlet and outlet hole on the outer wafers and a central wafer with I thin membrane that could be flushed with Hydrogen. See figure 5.2-1 below.





In a cross-section through the stack, figure 5.2-2 below, can the planned design be seen. The flat membrane has a central embossment with a small hole permitting gas to flow slowly from inlet to outlet and thereby exposing both sides of the membrane to Hydrogen, that situation can be kept

for a week or two nad when the membrane shall be tested is the inlet pressure rapidly increased causing the membrane to deflect until it hits the bottom wafer. The central hole is then sealed, if not leak tight, so at least to a level where the inlet pressure can be building up until the membrane bursts.



Figure 5.2-2 Sample Cross-section

5.3 Dynamic load test, bond interface

The quality of the bond burst test can be improved significantly if the samples are exposed to hydrogen under dynamic conditions i.e. large fluctuations in pressure or temperature prior to the burst. This could easily be done with a small modification of the upcoming Bond test equipment. If it is programmed to make a number of pressure cycles (say100 cycles to 80% of burst pressure) before going all the way up to burst. It could even stay for a while at load pressure between every load cycle.

5.4 Dynamic load test, mono crystalline Silicon

This is the most intriguing new test, the idea is to see if mono crystalline Silicon change it properties under high dynamic loads. The idea is to produce a number of samples, as depicted in figure 5.4-1 below. In a single silicon wafer a quite stiff beam is etched out with two comb actuator structures one on each side. Before the final etching the chip is mounted on a transparent carrier wafer. During the test is the free etched beam exitated in resonance by use of the two comb actuators and an optical feedback. After a burn-in time is the surrounding atmosphere changed to Hydrogen and the possible change in resonance frequency is monitored. The test chip is depicted in figure 5.4-1 below.





The basic idea is that if it is some cut-outs near the root of the beam it will be a point ot stress in which the silicon gitter is compressed or stretched out several thousand times per second, it would be a very sensitive indicator if something is going on i. e. if the material properties are changing in any way. In figure 5.4-2 is a simulation of a test beam depicted and it could be clearly seen where the stress concentration are located.



Figure 5.4-2 – Stress profile in the beam when the applied force is set to 0.1N. The maximum stress appears at the narrowing part of the beam and is simulated to $4.1E+8N/m^2$. This gives a safety factor (FOS) of 17. Due to that the maximum stress occurs at a processed surface the Yield strength should be lowered with approximated factor of 10. The estimated FOS is that case be around 1.7 which can be seen as the range where the beam could brake.

6 CONCLUSION HYDROGEN EFFECTS ON SILICON

The concept is a good example of the effect of "disruptive technologies". Advancements in one technical domain can suddenly generate a breakthrough in a completely different area. In this case, the system engineering progress in MEMS or Micro System Technology has generated a completely new approach for efficient hydrogen storage with an unprecedented storage capacity of 10-15 wt%.

The method to store in MacroSpheres is not limited to only Hydrogen, even if the most important gas, but any other gas can also be stored with the same method for other applications, such as a new type of diving suites or oxygen distribution to patients in hospitals, etc. Unfortunately is the technology behind the concept not very mature, so many aspects of the problem has to be investigated. The study presented in this report is a good step on the path towards a realization of the Hydrogen MacroSphere concept and has been giving many new insights in the problem. The EOARD office in London is gratefully acknowledged for the study.