

Innovative Silicon-Ceramic (SiCer) technology for high-strength pressure sensor applications using different manufacturing methods

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Abstract—A variety of manufacturing methods can be used to assemble silicon-based piezoresistive pressure sensors, such as anodic bonding or silicon direct bonding. To achieve high-strength pressure sensors, a high quality interface connection is required. The combination of silicon and ceramic creates a new innovative composite system known as Silicon-on-Ceramics, short SiCer. By using a special developed bondable Low Temperature Co-fired Ceramic tape, which corresponds to the thermal expansion coefficient of Si, the combination of both materials is possible through a sintering process. Advantages of the SiCer technology are high temperature stability, manufacturing at wafer-level and a high bond strength at the interface. Different manufactured SiCer-based pressure sensors were fabricated, electrically and mechanically characterized and compared with anodic bonded and silicon direct bonded pressure sensors.

Keywords—piezoresistive pressure sensor, SiCer, LTCC, ceramic, silicon, low pressure assisted sintering, burst pressure

I. INTRODUCTION

The production of piezoresistive pressure sensors divides into different steps, e.g. wafer processing including realization of measuring bridge and membrane as well as creation of an air channel through a back plate. Induced pressure through the channel results in a deformation of the membrane and the corresponding bending can be electrically evaluated via the measuring bridge. The connection of the wafer with the back plate can be achieved with various technologies, such as anodic bonding [1], [2] or silicon direct bonding [2], [3]. These methods are well established and easily accessible. However, with those manufacturing methods the achieved burst pressure measurements are in range of 100 – 200 bar.

The Silicon-Ceramic (SiCer) technology opens new possibilities for the fabrication of pressure sensors by combining the advantages of Micro-Electro-Mechanical-Systems (MEMS) and Low-Temperature Co-fired Ceramic (LTCC) technology. Characteristics of the SiCer composite substrate are high-strength bond connection at the interface depending on the used sinter method [4], temperature stability, individual pre-processability and reduced form factor. A comparison of different manufactured pressure sensors and the influence on the sensor thickness resulting from the respective back plate is shown in Fig. 1. Special manufactured LTCC tapes (bondable ceramic tapes - BCT, source: Fraunhofer IKTS), which are explicitly adapted to the expansion coefficient of Si, are used for the material composite of silicon and ceramics. The compound is achieved

in a sintering process; whereby a distinction is made between high pressure assisted sintering (HPAS), low pressure assisted sintering (LPAS) and other sintering processes such as a modified procedure based on tape-on substrate process.



Figure 1 Pressure sensor thickness in correlation to the manufacturing method (from left to right: anodic bonding, silicon direct bonding and SiCer technology)

II. FABRICATION METHODS FOR PRESSURE SENSORS

A. Previous manufacturing methods

Piezoresistive differential pressure sensors can be manufactured using various technologies, such as anodic bonding, silicon direct bonding or glass frit bonding.

Anodic bonding (AB) works without an intermediate layer by applying pressure, temperature and an electric field to the substrate to create a stable bond connection. In this process, the silicon wafer is bonded to a glass wafer containing alkali ions. Bonding takes place at temperatures between 300°C and 400°C and an additionally applied potential of 200 V to 1000 V. The connection is formed due to a thin SiO₂ layer at the bond interface, which is created by the reaction of oxygen on the Si-surface. [2]

With silicon direct bonding (SDB), two wafers are aligned with one another and initially pressed against each another. Adhesion is generated by hydrogen bonding and van der Waals forces. The resulting connection point continues to develop from the center over the rest of the area. A plasma-treatment of the Si-surface [5] enables low-temperature SDB at room temperature. The final bond strength is achieved through a tempering step at 200°C to 400°C, in which ionic bonds are formed between both Si-wafers [2]. This method does not require an intermediate layer to create a stable connection. [2], [3]

For glass frit (GF) bonding, a low melting glass paste is applied as an intermediate layer to a wafer via screen printing and bonded to a second wafer at temperatures of up to 400 – 600°C. During the bonding process, the glass paste is heated until it is liquid and the wafers are pressed together. When it cools down, the glass solidifies and a mechanically stable, hermetic connection is created. [2]

The authors thank the Federal Ministry of Education and Research (BMBF) for the financial support of this research project

B. Manufacturing process of SiCer substrates

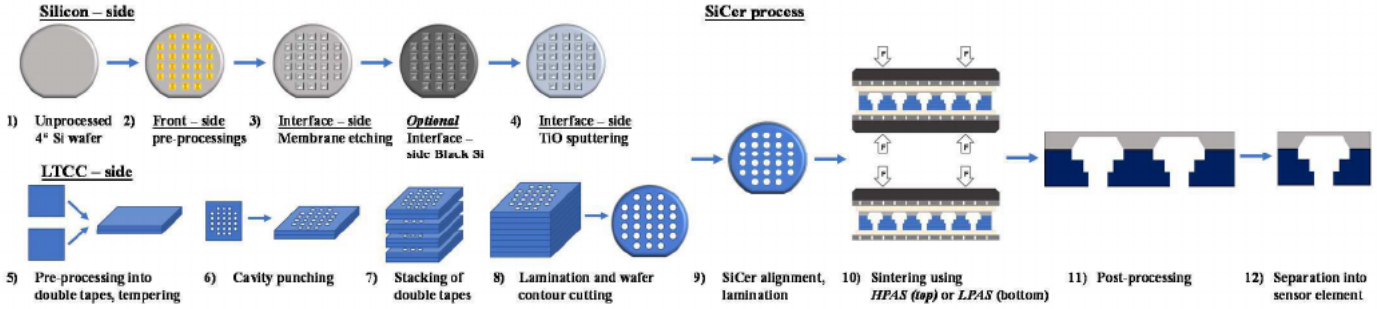


Figure 2 Schematically manufacturing process for SiCer-based pressure sensors

The SiCer process is divided into 3 main parts (Fig. 2), the respective/individual pre-processing of the Si side and the LTCC side as well as the SiCer combining/joining process.

A 4-inch Si-wafer (350 μm thickness, <100> crystal orientation, p doped) is used as the basis for the fabrication of the pressure sensor (1). Various processes such as lithography, etching and implantation are performed on the front side to achieve the functionality of the sensor (2). The later interface side is structured by etching with Potassium hydroxide solution (KOH) to generate 800 μm x 800 μm cavities with a membrane thickness of around 50 μm (3). Black silicon can be applied additionally onto the interface-side to enhance the bond strength [6]. To improve wetting [7] of the bond-interface a ~ 25 nm titanium is applied on the Si-wafer and thermally oxidized to ~ 50 nm thick titanium oxide. [4], [6]

The LTCC process begins with cutting the tape in the required processable size (115 mm x 115 mm) with consideration of the casting direction. This is necessary to compensate the casting direction related shrinkage deviation. Therefore, two BCT are stacked with a 90-degree rotation of the casting direction and isostatically pre-laminated with lamination parameter set (abbr. LPS): 1 (82°C, 10 MPa, 10 min process time including 4 min pre-heating) into a double tape layer stack (5). The created double tapes are tempered at 80°C for 10 min to drive out easily volatile solvents which stabilizes the stack for subsequent processing steps in the green state (no further shrinkage). The number of required tape-layers varies depending on the application. For the pressure sensor, 4 ceramic double tapes are necessary to create the channel structure. The cavities in the double tapes are punched in accordance with the respective layout (6). If required further pre-processing steps like screen-printing, via filling or laser structuring can be carried out. After pre-processing each double tape, the layers are stacked on a metal plate with stacking pins using alignment vias (7). A planar plate is placed on top of the ceramic to protect the cavities from deformation, followed by a silicone mat to smoothen sharp edges and thus allows an equal pressure distribution as well as prevents a tearing of the vacuum-sealed bag during lamination. The substrate is isostatically laminated with LPS: 2 (82 °C, 21 MPa, 15 minutes process time, including 4 min preheating) and a 100 mm wafer contour is cut out of the BCT laminate stack (8) with a picosecond laser (microSTRUCT C v2.0, 3D-Micromac AG). [4], [6]

In the next steps the pre-processed Si-wafer and Cer-laminate are joined together. Both materials must be precisely aligned to each other with a low lateral positioning tolerance (9). For this purpose, an optical-based adjustment and stacking machine is applied. Special structures on both materials are

used as alignment marks. After stacking of the Si-wafer and Cer-laminate a sacrificial High Temperature Co-fired Ceramic-tape (HTCC) is placed on top of the ceramic and isostatic lamination (LPS: 2) is used to create a temporary SiCer compound substrate. The HTCC sacrificial tape prevents the lateral shrinkage of the outer surface of the LTCC during the sintering process and is used as release layer to avoid sticking to the pressure plates later on. The final SiCer composite substrate is created by sintering (10), either HPAS (pressure range: 200 – 800 kPa, ATV PEO 603 sintering press) or with LPAS (pressure range: 1.8 – 15 kPa, ATV PEO 603). The Si-side is facing upwards in the sintering furnace to prevent the flow of the softening ceramic into the Si-cavities during the sintering phase. After sintering post-processing including cleaning steps, ultrasonic microscopy, X-ray examination and wafer-bow measurement at wafer-level (11) are carried out. Finally, the wafer is separated into individual 2 mm x 2 mm sensor elements using wafer sawing (12). [4]

III. SENSOR DESIGN, FABRICATION AND EVALUATION

A. Pressure sensor design and SiCer realization concept

A schematic structure of the pressure sensor with a straight cavity in the back plate is shown in Fig 3 a-c. For the presented investigations, a subdivision into different wafers is made: a) electrically functional wafers with a ~ 50 μm thick membrane, b) non-electrically functional wafers with a ~ 200 μm thick membrane and c) unstructured wafers without membranes. According to the respective experiments, a distinction between the 3 Si-wafer types is made. Unstructured wafers are used to verify the sintering profiles and to evaluate the bond-interface, non-electrically functional Si-wafers for application related burst pressure tests and electrically functional Si-wafers for electrical characterization of the sensor. Both Si-wafers with an etched membrane contain 966 individual sensor elements per wafer, each 2 mm x 2 mm in size with a bonding frame of ~ 3.5 μm .

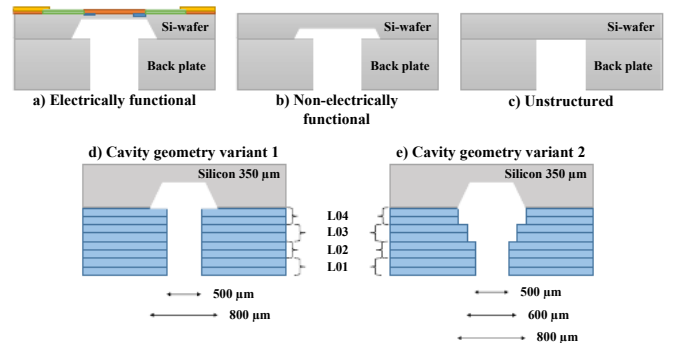


Figure 3 Pressure sensor structures (a-c) and approach to determine optimal cavity geometry in the ceramic back plate (d-e)

Previously only a straight cavity in the back plate could be examined due to the manufacturing method. LTCC multilayer technology and individual pre-processing of the ceramic tapes open up new possibilities for the back plate cavity. In addition to the straight ceramic cavity (Fig. 3 d, variant 1), a gradually widening cavity towards the Si-wafer (Fig. 3 e, variant 2) is examined and will be investigated in chapter III. B. The straight cavity consists of punched holes with a diameter of 500 μm , whereby for the gradually widening cavity 3 different cavity diameters 500 μm , 600 μm and 800 μm (from outer ceramic layer to silicon interface) were used. The cavity size in the layer L01 is predetermined at 500 μm , as this is the appropriate size for the burst measurement test station. The ceramic laminate for variant 1 was laminated in one process. For variant 2 a sequential lamination was initiated to ensure the stability of the varying cavity geometries. The L01 and L02 will first be laminated together using LPS: 2 (L012), afterwards L03 is stacked on top of the newly created L012 laminate stack and laminated using LPS: 1 (L0123). Separately the lamination of the Si-wafer and L04 is carried out with LPS: 1 (SiL04) and finally both individual created laminated parts (SiL04 and L0123) are combined using LPS: 2. Because of the different applied lamination pressures a stronger connection between the individual ceramic layers is achieved and the channel structure is strengthened for the following sintering process.

B. Manufacturing and evaluation of SiCer-based pressure sensors using HPAS process

The SiCer process was developed at Technische Universität Ilmenau and has been continuously advanced for more than 15 years [6], [8]. Due to the REACH-RoHS regulations, organic components of the SiCer-compatible ceramic tapes had to be replaced. This material change influenced the bonding properties between LTCC and Si. Therefore, the entire SiCer process had to be re-evaluated and optimized. The previously unsatisfactory bond-interface should noticeably improve by using HPAS. A first SiCer test with HPAS (Fig. 4) and the pressure sensor layout was performed. The optical inspection (a) as well as the bond-interface analysis (b) were satisfactory.

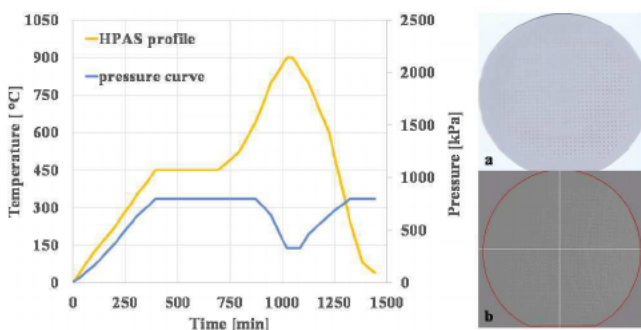


Figure 4 High pressure assisted sintering profile and resulting SiCer-substrate shown in a) optical inspection of the Cer-side and b) ultrasonic microscopy analysis of the SiCer bond-interface

Special non-electrically functional Si-wafers with a $\sim 200 \mu\text{m}$ thick membrane were used for this examination to determine the influence of the cavity geometry using burst pressure test. A machine-specific wedge error led to an uneven pressure distribution, which resulted in damaging $\sim 1/2$ of the sensor elements and thus reduced the yield of usable sensor elements. Nevertheless, SiCer sensor elements without visible impairments of each cavity variant were used for burst tests to evaluate this manufacturing method. During the burst pressure

test three fractures/events occurred: destruction of the sample (Si membrane or SiCer interfaces), testing up to machine limit of 400 bar without destroying the sample and other sources of error (e.g. detachment from the holder of the testing machine, not evaluated). With layout variant 1, bursting values of 335 – 400 bar (average: 378.5 bar and standard deviation: 18.7 bar) were achieved, whereas with variant 2 the results were in the range of 353.5 – 400 bar (average: 390.3 bar and standard deviation: 11.8 bar). The burst pressure test showed that a gradually widening ceramic cavity (variant 2) leads to slightly higher burst values as well as smaller scattering and will be exclusively used for all subsequent SiCer experiments.

Various artefacts were detected (Fig. 5) by optical inspection. The interaction of high temperatures and very high pressures creates dislocation lines in the Si (Fig. 5 – 1), deformations of the Si-membrane (2a) compared to an ideal Si-membrane (2b) and squashed/blocked ceramic channels (3 and 4). The occurrence of dislocation lines in the Si can lead to premature functional failures in the pressure sensor.

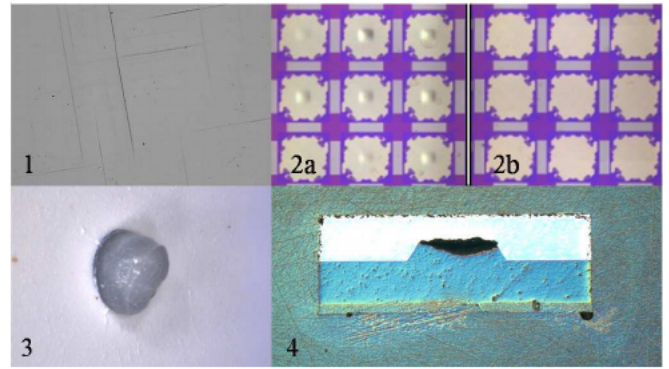


Figure 5 Imperfections found during evaluation of HPAS manufactured SiCer-based pressure sensors

C. Process adjustments and optimizations for LPAS method

Due to the mentioned imperfections of the manufactured SiCer-based pressure sensors, the sintering process was changed from HPAS to LPAS. For test purposes and because of cost reasons, unstructured Si-wafers without etched membranes were used for the sintering tests. The respective sintering results are shown in Fig. 6. A direct application of the HPAS temperature profile with a lower applied pressure resulted in noticeable cracking in the ceramic and a little to no bonded interface (Fig. 6 a).

A vast comprehensive process optimization of sintering temperature profiles was conducted during the project period [9]. Various factors that influence the sintering result were investigated, such as sintering temperature, heating- and cooling-rates, temperature plateaus and layout dependency. During the optimization, special attention was paid to: generating a fully bonded interface, crack-free ceramic layers depending on various layout designs as well as compatibility with different metallization pastes for soldering and wire bond applications. The detailed optimization will be presented in a separate work (in progress [10]). The developed sintering profiles (increased and optimized profile) were also tested with the pressure sensor layout.

By increasing the burn-out and sintering temperature by constant 40 K, cracking in the ceramic could be eliminated and the interface improved noticeably (Fig. 6 b). However, the very high density of cavities obviously still leads to delamination due to induced mechanical stress at the edges. For following investigations, the number of sensor elements

was reduced to 340 (Fig. 6 c), the number of BCT double tape layer were reduced from 4 to 3 and the temperature profile was changed to a more constant temperature gradient (Fig. 6, optimized LPAS profile – green curve). After successful manufacture, later increased to 460 and 586 (Fig. 6 d).

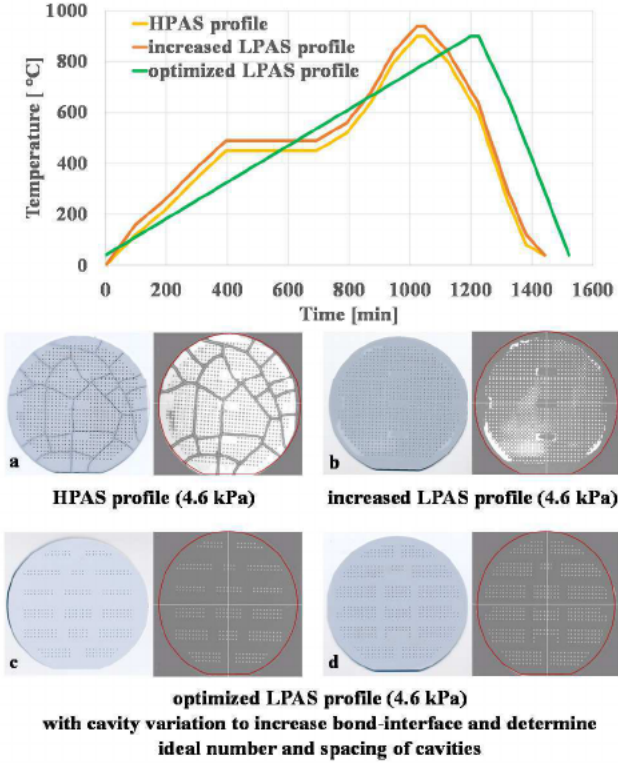


Figure 6 Process adjustment and layout variation of the ceramic cavity number to achieve a homogeneously bonded interface

D. Electrical characterization to examine the influence of the sintering temperature

The sintering tests have shown that increasing the temperature by 40 K leads to an improvement of the interface. Therefore, examinations with electrically functional Si-wafers (membrane thickness: $\sim 50 \mu\text{m}$) were carried out to determine the temperature influence on the pressure sensor's measuring bridge. For this purpose, standard pressure sensor samples (Silicon-Silicon, abbr. SiSi – SDB ref.) were manufactured using silicon direct bonding and electrically characterized to enable a realistic comparison. The Si part on which the measuring bridge is located was examined in 3 variants (blue-untreated/standard, green: tempered with 900°C and yellow: tempered with 940°C ; Fig. 7).

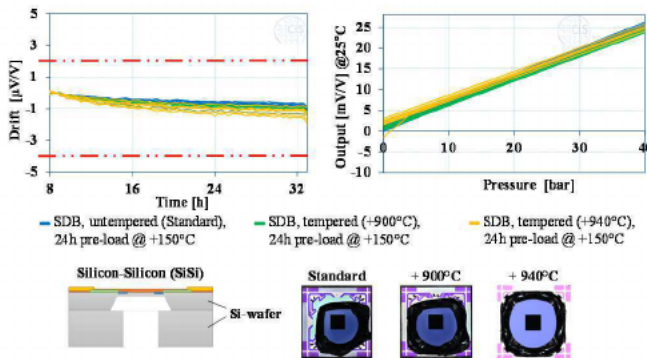


Figure 7 Electrical measurements of the zero point stability/drift with different temperatures using standard SiSi pressure sensor samples and respective burst fracture pattern of the Si-membrane

Based on these samples, electrical measurements at wafer level, testing of the bond interface using ultrasonic microscopy, pressure characteristics, sensitivity and hysteresis measurements, and burst pressure tests were carried out. It was found that the increase in the sintering temperature does not cause any electrical impairment of the sensor.

Finally, burst tests were carried out on the 3 pressure sensor samples to determine the influence of the temperature preload on the Si wafer. Values of 158 – 204 bar for the standard SDB, 157 – 203 bar for Si with a pre-load at 900°C and 145 – 160 bar for Si with a pre-load at 940°C could be verified. In all three variants, the membrane ruptured during the burst test, which indicates a stable connection at the bond interface. An influence of the sintering temperature at 900°C on the performance of the sensor cannot be detected either in the electrical measurements or in the bursting values. Using the increased sintering temperature shows no influence on the electrical functionality and a lower standard deviation was found in the bursting tests carried out, but the bursting values achieved are below the permissible limit of 160 bar.

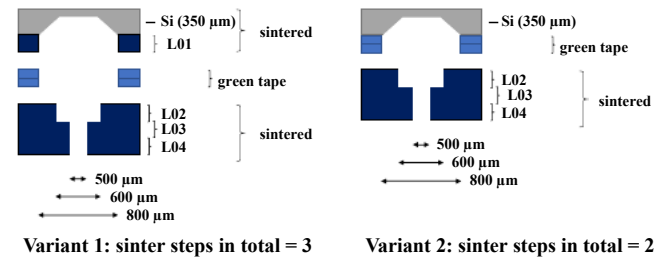
E. Modified SiCer approach: Substrate-on-Tape-on-Substrate (SoToS)

In order to maintain the yield of the sensor elements on the wafer, a modified process based on the tape-on-substrate method [11] was developed parallel to the sinter optimization. For the modified SiCer approach, two separately fabricated substrates were bonded with an additional unsintered ceramic layer by laminating and sintered a second time to form a SiCer substrate (Substrate-on-Tape-on-Substrate, SoToS). Two approaches were examined for the SoToS procedure (Fig. 8).

In variant 1, a BCT double tape is laminated (LPS: 2) to the Si wafer and then sintered, and the ceramic stack consisting of layers 1-3 is also sintered separately from the SiCer substrate. The final pressure sensor is then produced by laminating (LPS: 2) a green double tape between the two already sintered substrates and a third sintering process.

In variant 2, the ceramic part is sintered first, then the unsintered ceramic double tape layer is laminated (LPS: 2) directly to the interface of the two substrates and the final sensor is produced in a second sintering step. Both SoToS variants were first built with Si-wafers without cavities and tested for their suitability with the burst pressure test.

The results of the bursting tests were in average 188 bar (min.: 136 bar, max.: 202 bar) for variant 1 and 151 bar (min.: 54 bar, max.: 201 bar) for variant 2. A smaller deviation was noticed with variant 1 in comparison to variant 2. Based on the resulted measurements variant 1 was chosen for manufacture of a SiCer-based pressure sensor using a full functional Si-wafer.



Variant 1: sinter steps in total = 3

Variant 2: sinter steps in total = 2

Figure 8 Two approaches with the SoToS method to manufacture SiCer-based pressure sensors

IV. RESULTS AND DISCUSSION

After successfully examined implementation variants (chapter III.) electrically functional SiCer-based pressure sensors with a widening cavity in the ceramic back plate were manufactured:

- SiCer #1: HPAS, 800 kPa/ 260 kPa
- SiCer #2: HPAS, 800 kPa/ 260 kPa, pre-sawn Si
- SiCer #3: increased LPAS, 14.7 kPa, pre-sawn Si
- SiCer #4: optimized LPAS, 4.6 kPa and
- SiCer #5: SoToS variant 1, optimized LPAS, 4.6 kPa.

All pressure sensors (wafer-level) were first cleaned, the bond-interface optically inspected using ultrasonic microscopy, and precise positioning of both materials determined using X-ray analysis. The X-ray image (Fig. 9) shows the varying cavity diameters in the ceramic and the KOH-etched square cavity in the Si-wafer. The alignment was successfully performed and the ceramic cavities are centered under the Si cavity with a slight stacking misalignment.

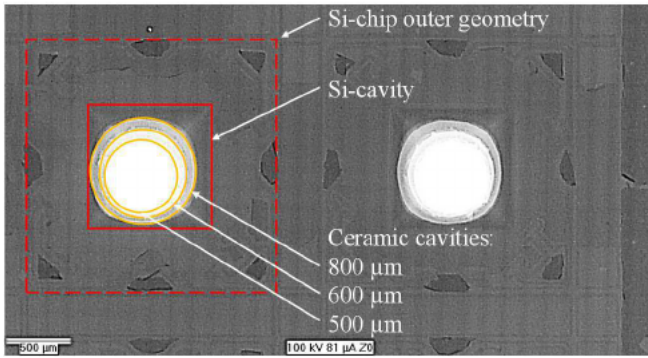


Figure 9 X-ray image of a SiCer pressure sensor after sintering process to evaluate the alignment accuracy

An important topic in MEMS sensor technology are the effects of aging, which directly affect the signal stability of the sensor systems. By measuring the zero-point stability (drift) of the piezoresistive test structures, the relaxation of stresses in the assembly process, e.g. due to creep or micro-crack growth, as well as the drifting of critical contact resistances or of the measuring resistors themselves, could be monitored with the highest application relevance. A quantitative reference level was achieved through the combination of thermal pre-aging. For the functional verification of the SiCer pressure sensor, the wafers were separated with a correspondingly adapted combined sawing process with a low chipping. The assembly was carried out onto special test sockets or evaluation boards, avoiding assembly stresses. The basic physical principles of the sensor and the processes as well as the properties of the SiCer material system and the possible mutual interactions were taken into account in the investigations. The electrical characterizations were carried out with a constant measuring current of 300 μ A. Fig. 10 shows the measuring curves of bridge resistance [k Ω m], sensitivity [mV/V], pressure characteristic [mV/V] and characteristic deviation [%FS]. All manufactured pressure sensor variants were electrically characterized. The illustration is limited to three selected pressure sensor variants; two SiCer pressure sensors (green: HPAS – SiCer #2; blue: LPAS – SiCer #3) and the standard SDB reference sensor (black – SDB ref.) without a temperature preload for comparison. The different measurement runs showed that the signal curves of the SiCer-based pressure sensors were comparable to those of the standard SDB sensor and are within the tolerance range. The adaptation of the

sintering temperature profile depending on the reduced sintering pressure resulted in a significant improvement in the sensor performance. The deviation of the non-linearity of both SiCer (#2 and #3) is negligible for this limit range.

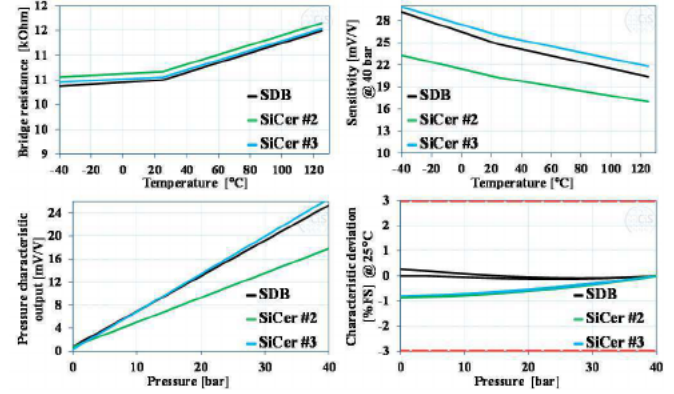


Figure 10 Measuring curves of the electrical characterization of the individual pressure sensors

The adhesion of the interface and the resulting burst strength of the assembled pressure sensors are tested with burst pressure method. All sensors were mounted onto special test specimens and subjected to pressures of up to 400 bar, which is the maximal capacity of the burst measuring station. For the pressure sensor a minimum burst value of 160 bar should be achieved in order to ensure the correct functioning of the pressure sensor. As reference, an anodic bonded pressure sensor, a standard SDB pressure sensor and glass frit bonded pressure sensors are included in Fig. 11 to allow a comparison and classification with the SiCer burst measurements. Predominantly a fracture in the membrane of the pressure sensor occurred and it can be assumed that the bond interface has higher strength than the membrane. A possible explanation for a membrane fracture could be previous damage to the membrane due to thermal pre-aging or process-related handling of the Si wafer, which induces tension. This would also explain why parts of the Si surface bursted as well. The GF #2 reference samples, which were single chip bonded achieved burst values over the higher burst values than GF #1 (wafer-level bonded), nevertheless both GF pressure sensors fractured at the bond interface and are not ideal for this specific pressure sensor manufacturing.

A significant influence of the sintering process in correlation of the resulting burst pressure measurements is visible. Both HPAS SiCer #1 & #2 samples showed very low burst values and the fracture occurred in the membrane. Because of high temperatures and high sintering weight during the sinter process various defects occurs and lead to premature failure of the Si membrane of the SiCer pressure sensors. SiCer #3 shows a rather large range with around 80 bar of the examined burst pressure and fractures occurred in the membrane again. SiCer #4 with the optimized sintering profile and a sintering pressure of 4.6 kPa reached burst values of 153 bar to 193 bar and the fracture occurred at the adhesive connection of the sample to the test specimen, which indicates that the actual burst strength of the composite pressure sensor could not be determined because of premature failure. Compared to the other pressure sensor samples where the fracture mainly occurred at the membrane, the SoToS (SiCer #5) sensors fractured within the ceramic. This indicates that the green tape layer, which is used as a bond layer between the two sintered parts, is the weakest part in the sample and it can be considered that the membrane is

apparently stronger than the ceramic and withstand burst pressures up to 193 bar. An influence of sintering temperatures and resulting stress on the membrane could not be proven with the burst pressure tests.

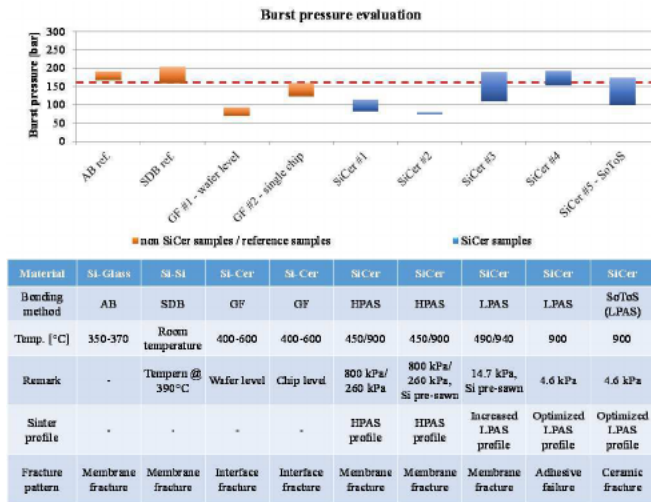


Figure 11 Comparison of different manufactured pressure sensors

V. CONCLUSION AND OUTLOOK

The measured burst values in this examination with differently produced SiCer-based pressure sensors showed a strong influence by the choice of the sintering process. HPAS (pressure range: 200 – 800 kPa) leads to imperfections in the sensor such as dislocation lines in the silicon and a deformation of the air channel as well as lower burst values because of the mentioned pre-damages of the membrane. The most promising burst values were achieved with low pressure assisted sintering (pressure range: 1.8 – 15 kPa) and the Substrate-on-Tape-on-Substrate (SoToS) process. LPAS manufactured SiCer pressure sensors achieved burst values around 110 – 193 bar. The SiCer SoToS method resulted in values of 99 – 174 bar, but the fracture took place inside the ceramic and not at the bond interface or the membrane as with the other sensors. SiCer #4 and #5 are comparable to the AB and SDB reference samples, which proves that the innovative SiCer technology can be used to manufacture high-strength pressure sensors. The advantage of the SiCer technology for pressure sensor applications is the possibility of individual designing of the ceramic cavity and therefore optimize the pressure distribution inside the sensor and increase the burst pressure resistance. Additionally, the overall thickness of the pressure sensor has been significantly reduced with the SiCer composite. Furthermore, depending on the application, an insulating capability of the pressure sensor may be preferred. SiCer-based pressure sensors possesses insulating capability due to the properties of the ceramic, whereas this can only be achieved with SDB sensors through additional steps.

Since the burst values of the SiCer SoToS approach showed very promising results, further examinations should continue to increase the bonding strength inside the ceramic layer, as the bond interface as well as the Si membrane withstood burst pressures up to 193 bar. An explanation for the partial disconnection within the ceramic layers can be found in the warpage of the ceramic part during the first sintering. To rule this theory out, tests with freely sintered ceramic parts are planned, whereby the shrinkage of the ceramic tape must first be determined in order to be able to adjust the layout accordingly. Furthermore, the process sequence with regard to the silicon side must be examined

more closely in order to avoid possible pre-damage of the Si membrane and thus further improve the bursting values of the Si membrane that have been achieved so far.

ACKNOWLEDGMENT

The authors gratefully thank the Federal Ministry of Education and Research (BMBF) for the financial support of the project (03WKDG03A, 03WKDG03E and 03XP0276D). Furthermore, we thank I. Koch and B. Müller (Electronics Technology Group, Technische Universität Ilmenau) for the technical support with the ceramic manufacturing and analysis of the substrates as well as D. Flock (Group of Materials for Electrical Engineering and Electronics, Technische Universität Ilmenau) for cross-section preparations. We also thank R. Grellmann and D. Hanig for the support with separation of the sensor elements, K. Pischek for SAM examination and analysis as well as S. Jagomast for carrying out the burst pressure measurements (CiS Forschungsinstitut für Mikrosensorik GmbH).

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