Silicon carbide membrane and new light source for a robust high temperature automotive pressure sensor

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Summary of experimental result direct bonding

In a previous study of the fiber optical pressure sensor[1], the bonding of two SiC plates to a membrane based on silicon carbide is explored using indirect bonding ("bonding with glue"). The "glue" involves constituents which are not silicon or carbon (i.e. part of silicon carbide), it is presented as mainly being a metal bonding. This indirect bonding is explored by Acreo.

Our approach is based on direct bonding. The motivation is that constituents other than silicon or carbon may cause problems (critical stress, long term use, etc.) due to differences in materials properties e.g. thermal expansion. Direct bonding would pave the way for a better lifecycle and a robustness since the constituents at the bonded interface are only silicon and carbon. In this pre-study, we explore the first steps toward a process for direct bonding of SiC plates ("bonding without glue"). The approach for this is surface engineering using a natural surface recrystallization process of silicon carbide. By that, improved bonding strength is expected to increase the membrane lifecycle and stability. The pre-study aim is to investigate if there are mechanisms which indicate a direct bonding between SiC samples the size of which is about 10x10 mm^2 using a new high temperature process. The exact details of mechanisms and uniformity over large area are not included in this work since it is a pre-study to motive larger efforts in full project.

We have developed high temperature (1600-2200^{o}C) SiC processes and methods since 1995 by various routes. We have also worked with producing epitaxial graphene on SiC with cavities[2] for a membrane structure for particle detectors. The cavities were made by Acreo. That experimental experience and collaboration are useful for studying the membrane which contains a cavity between two SiC plates.

Our literature study showed that only a few studies using non-SiC interfacial layer for bonding have been done. Direct bonding of SiC–SiC using surface-activated bonding applying an Ar ion beam was used to remove contaminants and oxide layer on the bonding surfaces[3]. The bonding was done at room temperature with 2.5 MPa pressure for 180 seconds. In that method, the preferential implantation causes problems due to the Si preferential sputtering during the SiC surface activation by the bombardment of an Ar ion beam. This has consequence that silicon atoms are removed from the SiC more than carbon atoms. A modified surface-activated bonding with a Si-containing Ar ion beam was attempted for the direct bonding of SiC–SiC, in which the Si-containing Ar ion beam was designed to realize compensation of Si loss. Still, the Ar ion beam causes crystal damage which makes membrane function not reliable and/or reproducible.

In comparison, our direct bonding approach is based on surface engineering[4] at high temperature which allows to explore a natural recrystallization process. There will be no oxide or contaminants since the surface is heated to high temperature and even thermally etched at

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[1] High temperature Fiber Optic Pressure Sensor-system, Swedish Energy Agency project P42093-1, 02/2016 – 04/2019.

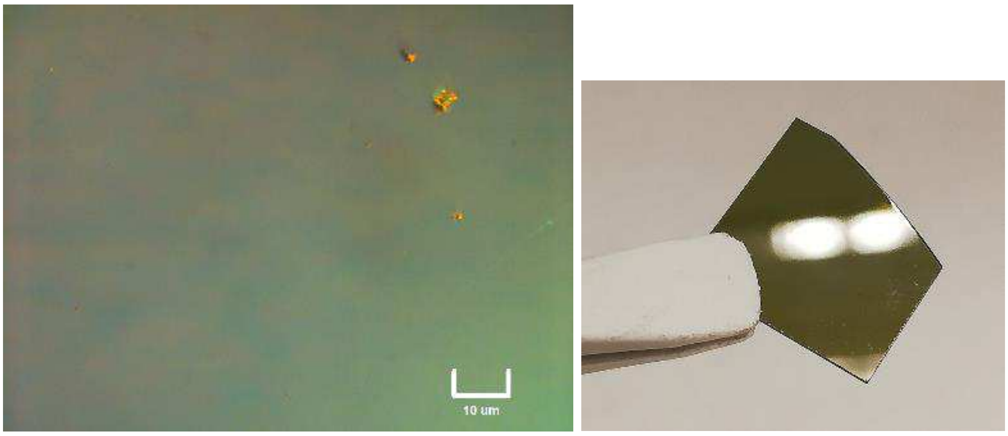
[2] Pallon, J; Syvajarvi, M; Wang, Q; Yakimova, R; Iakimov, T; Elfman, M; Kristiansson, P; Nilsson, EJC; Ros, L, Ion beam evaluation of silicon carbide membrane structures intended for particle detectors, NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH SECTION B-BEAM INTERACTIONS WITH MATERIALS AND ATOMS 371 (2016) 132-136

[3] A comparison study: Direct wafer bonding of SiC–SiC by standard surface-activated bonding and modified surface-activated bonding with Si-containing Ar ion beam; Mu et al; Applied Physics Express 9, 081302 (2016)

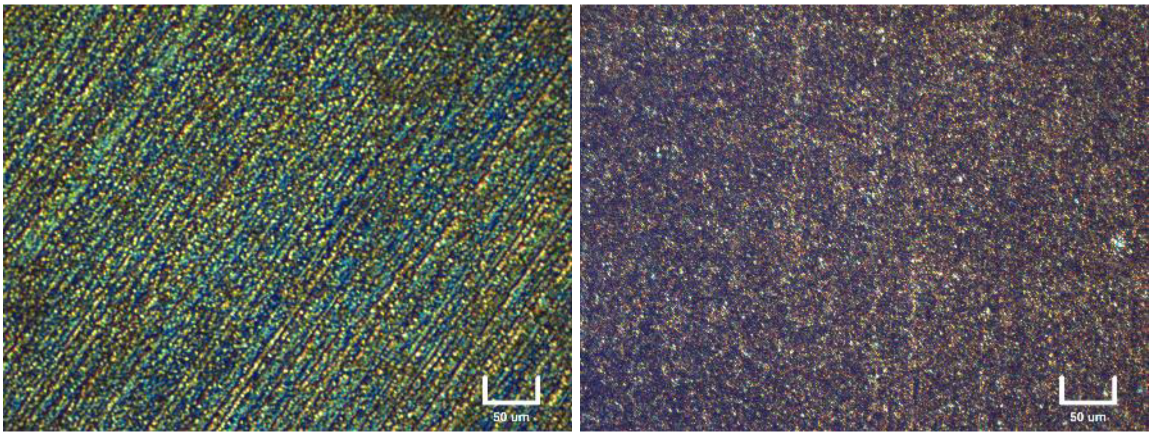
[4] Surface engineering of SiC via sublimation etching, Jokubavicius, Valdas; et al; Syväjärvi, Mikael, Yakimova, Rositsa, Applied Surface Science 390 (2016) 816

the initial stage. In this pre-study we have considered the following process related issues such as: Si/C ratio of sublimed species, ambient gas, heating elements, temperature (from 1600 to 1900°C). In addition, we have explored bonding of silicon carbide plates with different surface polarities (Si and C-face), surface structures, explore polycrystalline SiC, etc.

The experiments explore single crystal SiC with Si-face and C-face, and polycrystalline SiC (ceramic SiC). The single crystal SiC to single crystal SiC was explored to understand the surface behaviour since Si and C-face have different properties (for example surface free energy). The single crystal to polycrystal SiC was explored since it is closer to the actual need. The side facing the engine should not allow light to transmit since it will disturb the optical fiber signal, and polycrystal SiC is not transmitting light due to its irregular grain character. On the other hand, the polycrystal SiC is not flat and irregular grain structure which might not be part of surface recrystallization.

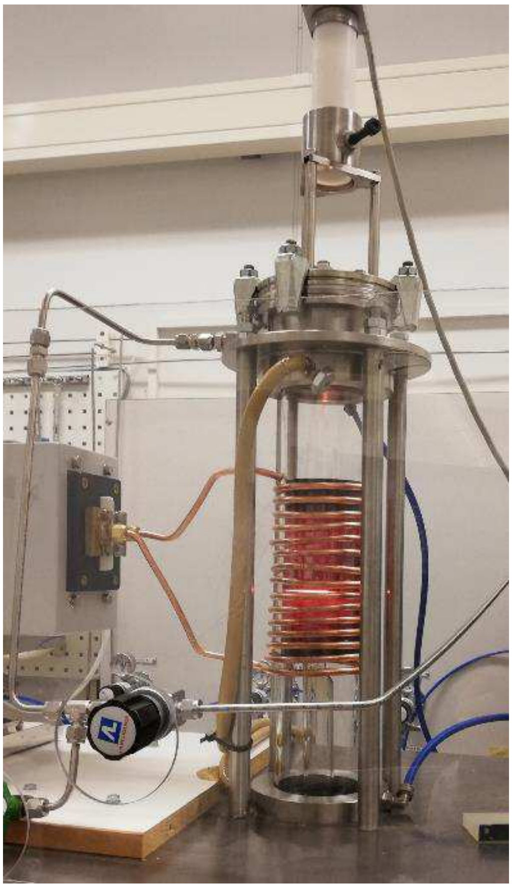


Left: optical micrograph of single crystal SiC sample. Right: overview of SiC sample and mirror like surface.



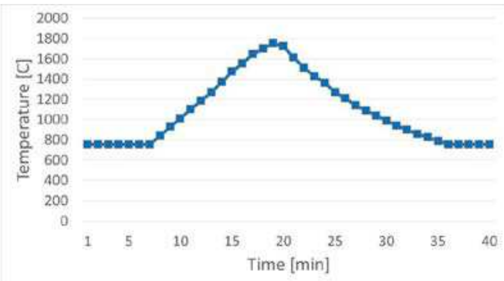
Optical micrographs of polycrystalline SiC sample surfaces (back and front with matt and rough polishing).

The heating furnace available for this project is an induction heating system. It is small size furnace which is readily available. Size is limited to 20 mm diameter samples. The radio-frequency generator and power matching unit are old (from early 1990s). It broke down at the start of the project. Replacement parts were tested but at the end we came to insight that it was not possible to repair. New RF generator was purchased. Breaking down of equipment and procurement process made delay in project, and project was extended.



Furnace using for direct bonding test

The graphite container geometry was modified to have fast heating and cooling rates. Fast and slow cooling is expected to relate to surface reconstruction, and thereby bonding. It is not clear if fast or slow cooling would be beneficial. In the experiments the fast cooling was applied. Once bonding mechanism is more mature, the influence of heating and cooling rates can be studied.



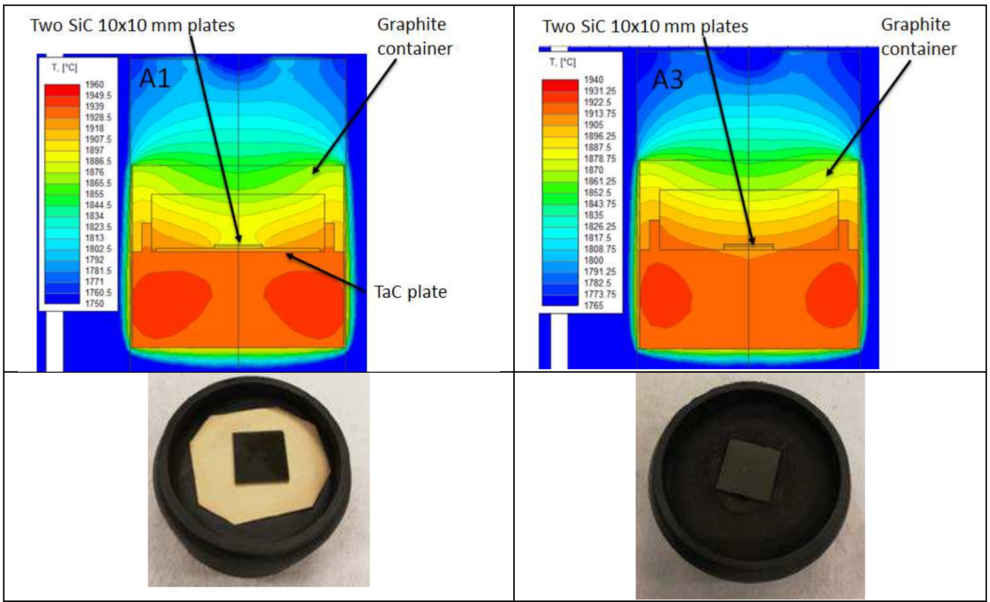
Two set of experiments were explored. First set in vacuum (0.1 mbar) at 1750-1850ºC range. Second set was at 2200ºC at 4 mbar range. At low pressure the vapor species can easier move, and the pressure range 1-5 mbar is well known to reduce vapor species transport[5]. The temperature is related to surface activity. The combination of vacuum and lower temperature, and higher pressure and higher temperature are parameter windows where careful surface activity is present and important for the maintaining of cavity in a full scale project.

[5] R. Yakimova, M. Syväjärvi, T. Iakimov, H. Jacobsson, P. Råback, A. Vehanen, and E. Janzén: "Polytype stability in seeded sublimation growth of 4H-SiC boules", J. Crystal Growth 217 (2000) 255

The test experiments indicate that Si-face is more active than C-face for bonding mechanism. This is likely due to that either Si leaves the surface before C, or that Si is active with C from the growth environment (graphite). The ratio of Si/C is known to be above 1 at high temperatures. The Si-face toward the polycrystalline SiC was also more active than C-face. This is probably a consequence that rough surface has initial sublimation or activity due to higher surface irregularity than a planar surface. The importance of Si is in line with the paper on modified surface-activated bonding with a Si-containing Ar ion beam for the direct bonding of SiC–SiC[3]. The non-regularity surface of using polycrystalline SiC is in line with need of argon sputtering, and it was also observed in early studies where the presence of a microscopic relief at the interface prevents the formation of gas traps, reduces the level of normal elastic stresses, and decreases the density of boundary dislocations in the regions between grooves[6].

The influence of Si/C ratio to the bonding was analyzed by using tantalum (Ta) in the bonding container and performing thermal simulations. In general, the Si/C ratio of sublimed SiC vapor species depends on temperature, but it can be modified by adding Ta in the container. Tantalum is a C getter, at elevated temperatures it reacts with carbon bearing species and forms TaC, in this way the Si/C ratio is modified. In addition, Ta couples well with the magnetic field and have different emissivity compared to SiC and graphite. Therefore, it can be used to change temperature profile and tune the Si/C ratio. The modeling of the temperature profile in the bonding container with and without Ta was done using a Virtual Reactor software which is used for modelling the growth of SiC crystals by sublimation technique.

Simple geometries were tested using Ta effect. The main insight is how using Ta affects the temperature gradient. According to simulations the axial temperature difference across the SiC plates when using Ta is about 7K, while without Ta it is only 1K. We did not observed any influence of Ta to sticking, this is probably related to the fact that we do not have a sticking mechanism to start from. However, the SiC samples annealed with Ta were free of surface graphitization at the same process conditions.

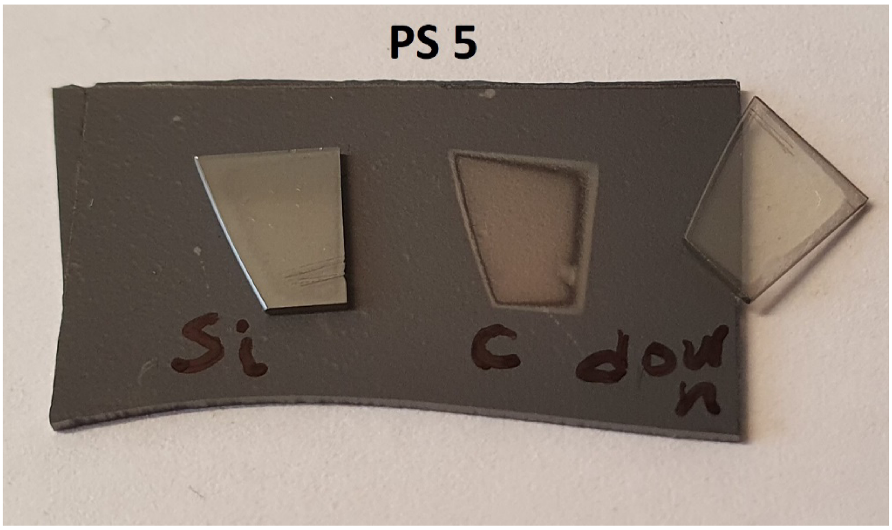


With Ta under the SiC	Without Ta

[6] Grekhov, I. V. et al. Direct bonding of silicon carbide wafers with a regular relief at the interface. Tech. Phys. Lett. 32, 453–455 (2006)

Only few samples have sticking of plates to each other. Some were possible to separate with surgical knife test (inserting knife blade between plates) or separated when dicing to cut into a cross-section to study interface. The mechanical testing was not taken further due to few samples, and no data indicating bond strength are available in this pre-study.

Sample with two sticking plates to each other and which separated while dicing.



Two samples of single crystal SiC with Si and C-face facing polycrystalline SiC. The sample with C-face facing SiC polycrystal did not stick, while Si-face sample did stick. The simultaneous experiment with both Si and C-face indicate Si-face as more active for direct bonding.