

Hydroxide catalysis bonding of silicon carbide

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Abstract

For bonding silicon carbide optics, which require extreme stability, hydroxide catalysis bonding is considered [Rowan, S., Hough, J. and Elliffe, E., *Silicon carbide bonding*. UK Patent 040 7953.9, 2004. Please contact Mr. D. Whiteford for further information: D.Whiteford@admin.gla.ac.uk]. This technique is already used for bonding silicate-based materials, like fused silica and Zerodur. In application with silicon carbide, the technique is highly experimental and the aim is to test the strength of the bond with silicon carbide. The silicon carbide is polished to $\lambda/10$ PV flatness and then oxidized at 1100 °C in a wet environment prior to bonding to form a necessary layer of SiO₂ on the surface. The bonding is performed in clean room conditions. After bonding the pieces are sawed into bars to determine the strength in a four-point bending experiment. The oxidization process shows many different color changes indicating thickness variations and contamination of the oxidization process. The bonding has been performed with success. However, these bonds are not resistant against aqueous cooling fluids, which are used during sawing. Several bars have survived the sawing and a maximum strength of 30 N mm⁻² has been measured.

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1. Introduction

Silicon carbide (SiC) has been used for structural applications since the 1960s. Because of its excellent performance in extreme conditions such involving abrasion, corrosion and high temperatures, it is now applied for fire bricks, heating elements and tubes, brake discs and seal rings for water pumps.²

In parallel with high temperature applications, the interest in SiC for application in completely different extreme environments is growing, e.g. in the space and semi-conductor industries. These applications require extreme shape stability in vacuum (and possibly cryogenic) environments. In the space industry the main applications have been for mirrors and some support structures for those mirrors. Examples of SiC mirrors that are already in an Earth orbit are the Narrow Angle Camera of Rosetta and mirrors for Rocsat 2.³ GAIA is a future mission of which nearly the entire payload will be constructed of SiC.⁴ The payload will contain a laser metrology system, which con-

sists of a number of SiC optical components, mounted onto a SiC optical bench. The positional stability required of the optical components relative to each other is 0.19 pm over a period of 6 h. This requires a joining technique for SiC that does not introduce stresses and is very stable. Hydroxide catalysis bonding can be of interest for this specific application.¹

Hydroxide catalysis bonding or ‘silicate’ bonding is a bonding technique invented and patented by Gwo^{5,6} at Stanford University. The technique has been used in the Gravity Probe B space experiment (successfully launched in 2004). The technique has been applied by the Institute of Gravitational Research at the University of Glasgow in the GEO 600 gravitational wave detector⁷ and will be applied on the LISA Technology Package interferometer for LISA pathfinder.^{8–10} It has however not thoroughly investigated for bonding SiC.

The goals of the hydroxide catalysis bonding experiments which are discussed in this paper were threefold:

- gain experience in polishing SiC to $\lambda/10$ PV flatness;
- gain experience in bonding SiC with the hydroxide catalysis bonding technique; and

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- determine the strength of the SiC–SiC hydroxide catalysis bonds in air at ambient temperature.

The main focus however, is on gaining experience in bonding SiC.

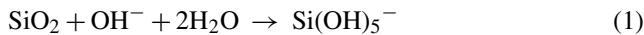
2. Hydroxide catalysis bonding technique

The hydroxide catalysis bonding technique is a technique that achieves bonding between a number of materials if a silicate-like network can be created between the surfaces, or in other words any silica containing material. Examples are silica, Zerodur, fused silica, ULE glass and granite.

The two silicate-based materials are bonded using an alkaline bonding solution: like sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium silicate (Na₂SiO₃) dissolved in water.

The bonding surfaces must have a peak-to-valley (PV) flatness of ≤ 60 nm if a hydroxide solution is used. Both bonding surfaces are cleaned in a clean environment to be free of chemical and particulate contaminants. The bonding solution is filtered and dispensed on the bonding surface with a volume of $\geq 0.4 \mu\text{l}/\text{cm}^2$. The other substrate is then placed gently on top of the substrate with the bonding solution and can be compressed slightly to ensure a uniform bond. At this moment the hydroxide catalysis commences and consists of three steps⁵:

1. *Hydration and etching*: in which the OH⁻ ions in the bonding solution act as a catalyst and etch the silica surfaces in contact. This causes the liberation of silicate ions:



2. *Polymerization*: due to the hydration the active number of OH⁻ ions reduces and the pH of the solution decreases. If the pH < 11, the silicate ions dissociate:



And siloxane chains and water are formed:



Once the siloxane chains are formed the bond is rigid.

3. *Dehydration*: in which the water migrates or evaporates. After 4 weeks of curing at room temperature full strength is achieved.

The time taken for a bond to form can be controlled by a combination of temperature and pH of the bonding solution used.¹¹ The bonding thickness is approximately 50 nm. The roughness is not an issue. The roughness can even be 0.5 μm to avoid optical contacting during alignment.

SiC cannot be used directly for hydroxide catalysis bonding. During polishing to $\lambda/10$ PV flatness, any SiO₂ layer formed during sintering is removed. To make bonding of to SiC components possible, the surface must have a thin layer of SiO₂. This layer is formed after cleaning the SiC pieces and then placing the

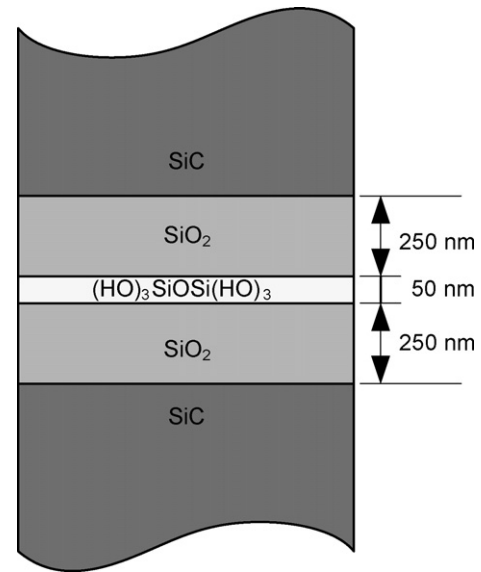


Fig. 1. Layer structure of hydroxide catalysis bonded SiC pieces.

pieces in a quartz tube furnace at 1100 °C in a wet nitrogen environment. The SiO₂ layer thickness must be smaller than 250 nm to maintain the $\lambda/10$ PV flatness.

The layered structure of hydroxide catalysis bonded SiC pieces is shown in Fig. 1.

3. Approach

The hydroxide catalysis bonding experiments consisted of seven steps, each of which is discussed in this section:

- sawing SiC blocks;
- polishing the SiC blocks;
- oxidization of the SiC blocks;
- hydroxide catalysis bonding of the SiC blocks;
- sawing bars from the bonded SiC blocks;
- viewing bonds under the scanning electron microscope (SEM); and
- four-point bending experiments on SiC bars.

3.1. Sawing SiC blocks

First, blocks were sawed from three different types of SiC (Boostec SSiC with and without CVD SiC coating, Xycarb C/SiC with CVD SiC coating and Hexoloy SA SSiC without SiC coating). For a representative measurement of the bending strength at least 25 bars/material should be tested, such that a reliable statistical analysis can be performed. Because the HCB technique is an experimental technique as well, enough surface area had to be bonded to make 50 bars. The dimensions of the blocks are shown in Table 1.

3.2. Polishing SiC blocks

The bonding surfaces were polished to $\lambda/10$ PV flatness. To achieve this flatness, the blocks of Boostec material and Xycarb

Table 1
Dimensions of the blocks

Material	Number of blocks	<i>h</i> (mm)	<i>b</i> (mm)	(1/2) <i>l</i> (mm)
Boostec SSiC + CVD SiC	8	35.0 ± 0.2	10.5 ± 0.2	22.5 ± 0.2
Boostec SSiC + CVD SiC	8	35.0 ± 0.2	10.5 ± 0.2	22.5 ± 0.2
Boostec SSiC	8	35.0 ± 0.2	10.5 ± 0.2	22.5 ± 0.2
Boostec SSiC	8	35.0 ± 0.2	10.5 ± 0.2	22.5 ± 0.2
Xycarb C/SiC + CVD SiC	8	35.0 ± 0.2	10.5 ± 0.2	22.5 ± 0.2
Hexoloy SSiC	6	6 equal (±0.5 mm) wedges from a Ø76 mm disc		13.0 ± 0.2

material were bonded with an adhesive onto one pan as shown in Fig. 2a. The Hexoloy blocks already formed a ring, and were polished on a separate pan (Fig. 2b). The polishing was conducted with 3 µm diamond powder type O of Kemet on a siphon machine with 38 rotations/min and 3 kg load for 80 h.

3.3. Oxidization of SiC blocks

After ultrasonic cleaning in an acetone bath, the blocks were oxidized in a quartz tube oven at 1100 °C in an oxygen deficient environment. This environment was created by bubbling zero-

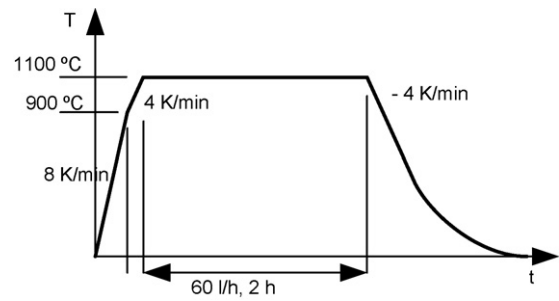


Fig. 3. Oxidization temperature scheme.

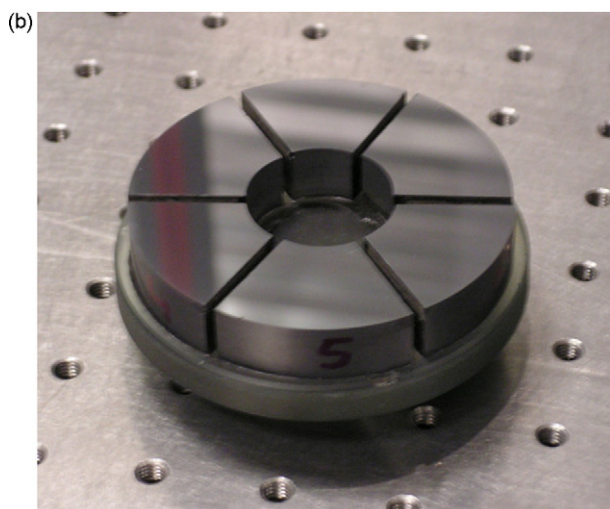
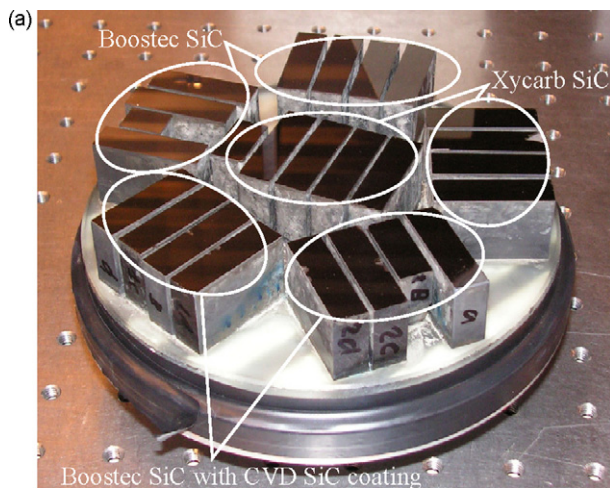


Fig. 2. Polishing pans with (a) Boostec and Xycarb SiC and with (b) Hexoloy SiC.

grade nitrogen through demineralised water at 80 °C. This wet nitrogen mixture was pumped through the oven. The oven heating scheme is shown in Fig. 3. The bubbling was initiated slowly at 900 °C. At 1100 °C the flow is increased to 60 l/h. This flow level and temperature was maintained for 2 h. The oxidation set-up is shown in Fig. 4.

3.4. HCB bonding

The blocks were bonded using the HCB technique. Prior to bonding the blocks were thoroughly cleaned with cerium oxide and sodium bicarbonate powder in de-ionised water to make the bonding surfaces hydrophilic. The bonding was performed in clean room conditions by mixing a sodium silicate solution (14% NaOH and 27% SiO₂) with de-ionised water with a volume ratio 1:6. The bonding solution was applied in the volume of 0.4 µl/cm² to one of the bonding surfaces upon which the other bonding surface was placed. The sodium silicate, the water

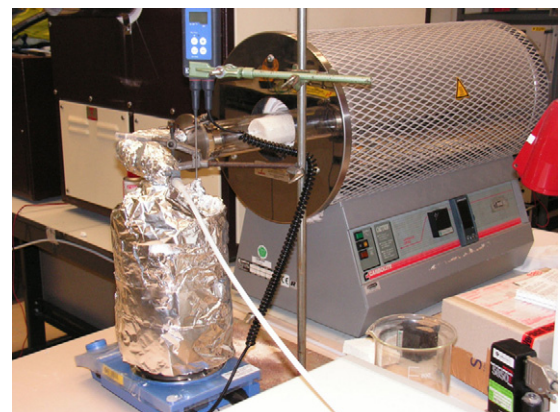


Fig. 4. Oxidization set-up.

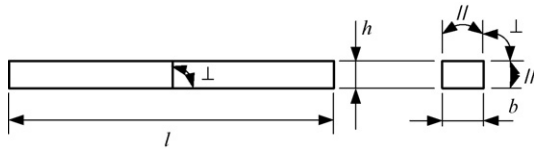


Fig. 5. Dimensions of a bar.

and the silicon oxide on the bonding surfaces immediately form siloxane chains bonding the two surfaces together. The bonds were made partly at the University of Glasgow, Department of Astronomy and Physics. The other bonds were made at TNO Science and Industry in Eindhoven. The bonds were cured in air for 3 weeks, 1 week of which the bonds were held at 50 °C.

3.5. Sawing bars

The blocks were sawed and ground into bars, such that the HCB bond was in the centre as shown in Fig. 5. The desired dimensions of the bars for each material are shown in Table 2. However, the final dimensions were dependent on the success during sawing.

3.6. Viewing bonds with the SEM

The bonds were inspected under the SEM after sawing to assess the SiO₂ layer thicknesses and the HCB bonding thickness.

3.7. Four-point bending experiments

The bars were subjected to a four-point bending experiment according to ASTM norm C1161-2C.¹² A schematic illustration of a four-point bending set-up is shown in Fig. 6.

The ASTM norm is focused on determining the bending strength of ceramic materials. In the four-point bending experiment the force F was increased slowly with prescribed crosshead speed of 0.55 mm/min. The applied force and crosshead speed were measured. Between the upper two rods the moment along the bar is constant, and thus the stresses over distance $L/2$. The maximum stress upon fracture during the bending experiment was calculated using¹²

$$\sigma_{\max} = \frac{3FL}{4bh^2} \quad (4)$$

Table 2
Bars with dimensions and tolerances according to the ASTM norm (use Fig. 5 for understanding of the symbols)

Material	h (mm)	b (mm)	l (mm)	\perp (mm)	\parallel (mm)
Boostec + CVD	3.00 ± 0.13	4.00 ± 0.13	45	0.015	0.015
Boostec	3.00 ± 0.13	4.00 ± 0.13	45	0.015	0.015
Xycarb + CVD	3.00 ± 0.13	4.00 ± 0.13	45	0.015	0.015
Hexoloy	1.58 ± 0.07	2.1 ± 0.07	26	0.015	0.015

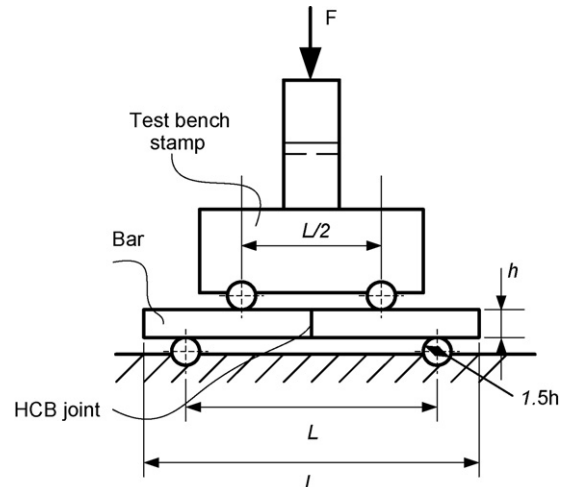


Fig. 6. Schematic representation of the four-point bending set-up.

4. Results

For all experimental steps, the results and observations are discussed in this section.

4.1. Polishing the blocks

After polishing for 1.5 weeks the required flatness of $\lambda/10$ PV or 0.1 waves was obtained for all Hexoloy blocks and all but one Xycarb blocks. The flatness achieved for the Boostec blocks was in most cases worse than 0.1 wave (Table 3). The largest deformation of the surfaces was seen at the long edges of the polished surfaces. The surfaces of Boostec SSiC and Xycarb C/SiC are generally convex.

The Xycarb blocks mounted in the centre were slightly flatter on average than the Xycarb blocks on the outer part of polishing pan.

4.2. Oxidization

The blocks were oxidized in four sessions. In the first two sessions the coloration of the polished surface due to oxidization showed a dependency of the position in the oven. In the front of the wet air flow the discoloration is less than further in the flow. This has, however, not been observed in the last two sessions.

In the first two sessions, the color change of the Xycarb and Hexoloy blocks was yellow (Fig. 7a) and the color change of Boostec material was increasingly orange to blue (Fig. 7b). However, in the last two sessions the discoloration of all materials varied from yellow to purple to blue. Some blocks showed small

Table 3
Measured flatness in waves

Block	Overall flatness (waves)	<0.110 wave?
Boostec SSiC + CVD SiC		
UAcvd1	0.119	No
UAcvd2	0.077	Yes
UAcvd3	0.164	No
UAcvd4	0.159	No
UAcvd5	0.130	No
UAcvd6	0.089	Yes
UAcvd7	0.103	Yes
UAcvd8	0.148	No
Boostec SSiC		
UA1	0.127	No
UA2	0.088	Yes
UA3	0.161	No
UA4	0.151	No
UA5	0.152	No
UA6	0.095	Yes
UA7	0.134	No
UA8	0.155	No
Xycarb C/SiC + CVD SiC		
UB1	0.105	Yes
UB2	0.088	Yes
UB3	0.061	Yes
UB4	0.096	Yes
UB5	0.115	No
UB6	0.109	Yes
UB7	0.076	Yes
UB8	0.104	Yes
Hexoloy SSiC		
UC1	0.098	Yes
UC2	0.099	Yes
UC3	0.097	Yes
UC4	0.092	Yes
UC5	0.099	Yes
UC6	0.098	Yes

spots of different color (Fig. 7b). The Hexoloy blocks showed the most uniform color changes (Fig. 7c). Finally, marker residuals to indicate the nonflat parts of the reflective surface, which have visually been removed during cleaning, became visible again after oxidation (Fig. 7d).

4.3. Bonding the blocks

Of each material, one half was bonded in Glasgow and the other half was bonded in Eindhoven in the interest of transfer of knowledge from the University of Glasgow to TNO in Eindhoven. The bonding has been performed successfully for 13 of 15 bonds. The two remaining bonds (both made in Eindhoven) proved unsuccessful after 3 weeks curing (Table 4). The failure of these bonds was attributed to dust and a hair on the bonding surface, which prevented good bonding. There were some small differences between the bonding in Glasgow and Eindhoven:

- In Glasgow, the blocks were kept wet after cleaning and have been wiped with methanol just before bonding because they had to be transported from a cleaning facility to a clean room facility. In Eindhoven, the blocks were cleaned in clean room

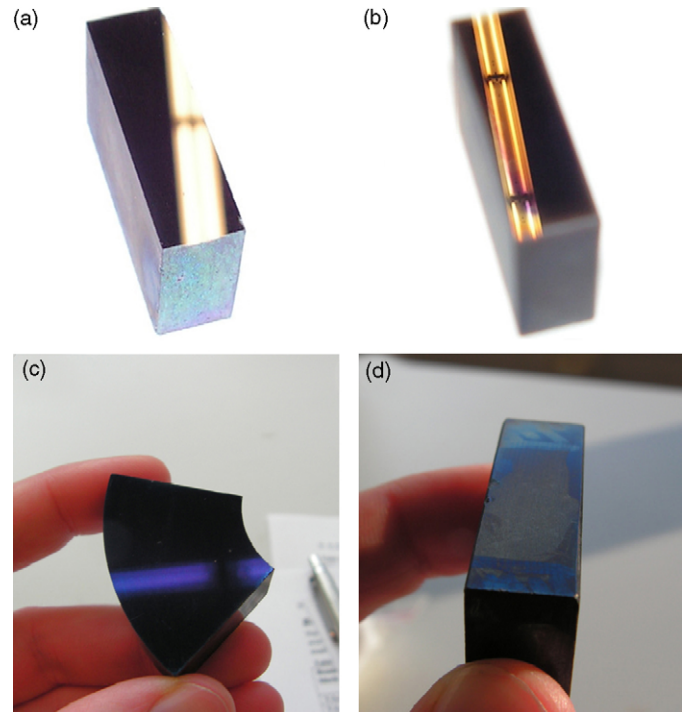


Fig. 7. Some examples of oxidized blocks (a) yellow on Xycarb C/SiC with CVD SiC, (b) orange to purple on Boostec SSiC with CVD SiC, (c) uniform blue on Hexoloy SSiC and (d) marker residuals on Boostec SSiC with CVD SiC. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

conditions and have been blown dry with clean air. The blocks have not been wiped with methanol.

- In Glasgow, the bonding solution was filtered. In Eindhoven the bonding solution was not filtered because filters were unavailable.
- In Glasgow the bonds were made with exactly 1.4 μl bonding solution for the Boostec and Xycarb blocks and with 2.4 μl for the Hexoloy blocks. In Eindhoven all bonds were made with $\sim 5 \mu\text{l}$ bonding solution (Table 4).

Table 4
Blocks bonded with bonding time and the amount of bonding fluid used

Bonded block	Block 1	Block 2	Location	Amount of solution (μl)	Successful
VAcvd1	UAcvd6	UAcvd7	Glasgow	1.4	Yes
VAcvd2	UAcvd4	UAcvd8	Glasgow	1.4	Yes
VAcvd3	UAcvd3	UAcvd3	Eindhoven	5	Yes
VAcvd4	UAcvd1	UAcvd5	Eindhoven	5	No
VA1	UA4	UA8	Glasgow	1.4	Yes
VA2	UA3	UA5	Glasgow	1.4	Yes
VA3	UA1	UA6	Eindhoven	5	Yes
VA4	UA7	UA2	Eindhoven	5	Yes
VB1	UB1	UB2	Glasgow	1.4	Yes
VB2	UB3	UB4	Glasgow	1.4	Yes
VB3	UB5	UB6	Eindhoven	5	Yes
VB4	UB7	UB8	Eindhoven	5	No
VC1	UC1	UC4	Glasgow	2.4	Yes
VC2	UC2	UC6	Glasgow	2.4	Yes
VC3	UC3	UC5	Eindhoven	5	Yes

4.4. Sawing bars

The sawing of the blocks which was largely unsuccessful. Initially, bonded blocks – VAcvd1, VAcvd2, VA1, VA2, VB1 and VB2 – were sawed simultaneously into bars with dimensions: 45 mm × 10.5 mm × 3.2 mm. The bars were sawn with a speed of 8 μm/stroke. After removal from the pan by heating the resin, only 16 bars had survived the sawing of which 7 and 8 bars came from bonded block VA1 and VB2, respectively. All other bars had failed on the bonding surface.

The broken bars were cleaned with alcohol to remove the resin. Next, the bonding surfaces were inspected. Most surfaces appear to have only the SiO₂ layers (Fig. 8a). Residuals of the siloxane bond layer did not appear to be present, except on the bars of VB1 and VAcvd2. On them, some small bubbles were visible (Fig. 8b).

4.5. SEM inspection

A photograph was taken with a SEM of the bond layer of bar from VB2, which survived the sawing (Fig. 9). The sample has not received any additional treatment (like polishing) to get an optimal image. Because of this and because the bond is not present along the edges, the thickness of the bond layer including the SiO₂ layers, could only be estimated at 260 nm. This thickness is in the same order of magnitude as the expected thickness, which was less than 550 nm.

4.6. Four-point bending

Due to the fact that the sawing was not totally successful, the four-point bending experiments have not been conducted completely according to the ASTM-norms. The experiments have been performed on 11 bars with a 10 kN tensile testing bench with contact cylinders of Ø3 mm.

The conditions during measurements were

1. room temperature: 20 °C;
2. outside air pressure: 1003 mbar;
3. $L = 40$ mm;
4. $l = 20$ mm;
5. crosshead speed: 0.5 mm/min;

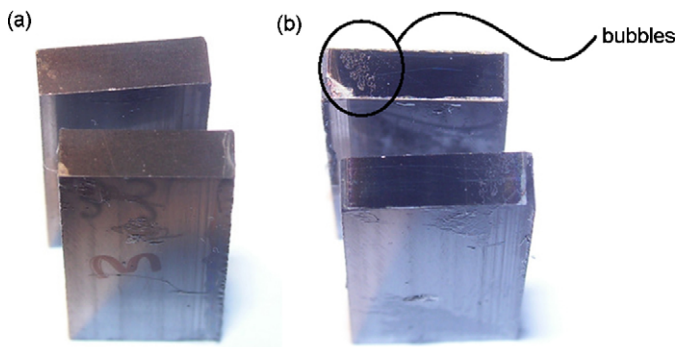


Fig. 8. Bonding surfaces of samples that failed during sawing (a) a bar from block VA2 and (b) a bar of block VAcvd2.

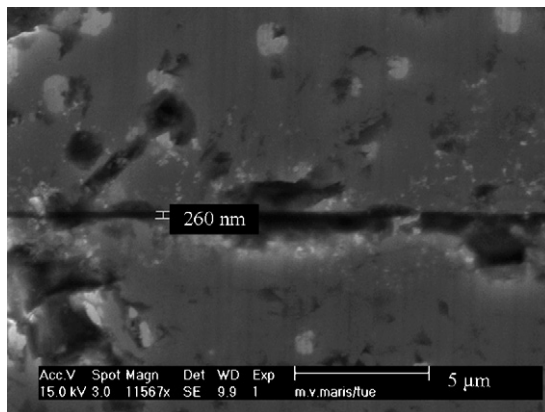


Fig. 9. SEM image of the bond layer of a bar from block VB2.

6. the bars have dimensions: 45 mm × 10.5 mm × 3.2 mm; and
7. eleven bars have been broken.

The results of the experiments are shown in Table 5. The force–time diagrams for two specimens are shown in Fig. 10. The table shows maximum forces that have been measured for the bars from VA1 with an average maximum force of 103.9 N and a maximum deviation of 7.4 N. This is equivalent with an average strength of 29.3 N mm⁻² provided that 100% of the surface has bonded successfully. The maximum forces that have been measured for the bars from VB2 seem to be divided into two groups: one with average 23.1 N and one with average 48.6 N corresponding with 6.3 N mm⁻² and 13.3 N mm⁻², respectively.

When viewing Fig. 10 one can see that the force does not increase smoothly. This is due to the fact that the specimens have not been ground to remove standing edges in fear of fracture of the bond during grinding. These standing edges fracture first, resulting in a sudden reduction in measured force. The final reduction of the force shows the fracture of the bond.

5. Discussion

Every step of the HCB bonding of SiC has been an experiment. This leads to a large number of uncertainties regarding the

Table 5
Results of the bending experiments

Bonded block	Bar	Maximum force (N)	Maximum stress (N mm ⁻²)
VB2	22	23.0	6.3
VB2	23	26.1	7.6
VB2	24	20.2	5.4
VA1	62	109.6	30.7
VA1	63	105.8	31.5
VA1	64	96.4	27.0
VB2	25	57.8	15.9
VB2	26	41.1	11.0
VB2	27	46.9	12.9
VA1	65	97.6	27.3
VA1	66	109.8	29.8

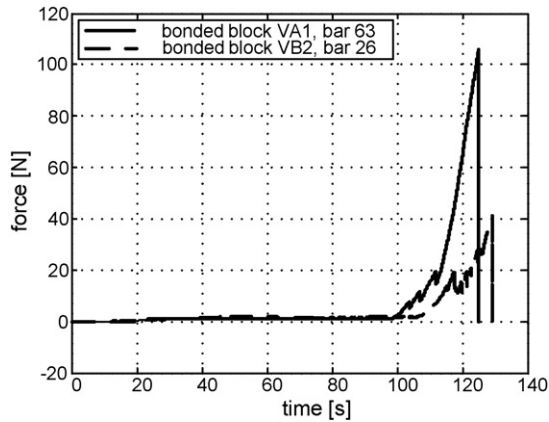


Fig. 10. Force–time diagram of two samples.

fracture of most bars during sawing and measuring the strength of HCB SiC–SiC bonds:

- The bonding surface flatness achieved was not in all cases the desired $\lambda/10$ PV due to the difficult geometry of the samples. This may have contributed to bonds that are successful over only part of the bonding surface.
- The bonding surface flatnesses achieved, showed different values for the different types of SiC. It is not clear why the flatness of the Boostec blocks was lower than the Xycarb blocks. A possible reason for this might be the fact that Xycarb SiC has a lower Young's modulus. Although the stiffness of the Hexoloy SiC is approximately equal to the Boostec SiC stiffness, better flatness has likely been achieved because of the more circular polishing pan geometry.
- The oxidization process was not very well controlled. It appears that there is a large dispersion of the SiO₂ layers and that the formation of the layers using the wet-oxidization process is sensitive to contamination. This technique has been used because the SiO₂ layer have grown in situ from the SiC, making the SiO₂ bond to SiC most probably much stronger than the HCB bond between the SiO₂ layers. However, creating a SiO₂ layer using chemical vapour deposition is thought to be weaker but is also considered to be much better controllable. This means that CVD SiO₂ can be applied with a controllable and more even thickness, and the process is less sensitive to contamination.
- The bonding process of the 13 blocks that have been bonded successfully, are not a complete success in the sense that the bond may well have occurred over less than 100% of the bonding surface, due to a lack of flatness of the bonding surfaces, due to contaminations or due to a lack of bonding solution in the interface. The blocks may not have bonded over 100% of the surface due to the latter argument.
- The differences in the bonding process may have caused differences in the bonds. The bonds made in Glasgow, used filtered bonding fluid to ensure no particulates larger than 0.2 μm in diameter were present in the fluid and the volume

of solution used was more carefully controlled. This might have led to a higher success rate of the bonds.

- The fracture of the bonds during sawing can be explained by each of the arguments above in combination with two possible properties of the sawing process:
 1. During sawing forces are applied to the samples, which cause stresses larger than the strength.
 2. The cooling fluid (95% water) attacks the bond. The argument is that free OH⁻ ions in the water have a tendency to reverse the formation of siloxane chains, thereby reducing the strength of the bond. To test this, four samples that have survived sawing initially, have been subjected to the cooling fluid (pH 7.8) and to a buffer with a pH of 4.1 for 12 h. After this period the bonds failed at the slightest handling. It is recommended that sawing forces are reduced by using a thinner sawing blade and by reducing the sawing speed and to use an oily cooling fluid.
- The goal of measuring the bond strength has not been met entirely, because only 11 bars could be tested in total, instead of the intended 25/SiC type. Also because only 11 samples have been tested and because there are too many uncertainties regarding the true bonding surface area, the measured strengths of the bonds in a range of 5–30 N mm⁻² cannot be subjected to any statistics. What can be said is that the maximum strength of ~ 30 N mm⁻² is encouraging. The measured maximum strength is in the region of typical epoxy adhesive bond strengths, which is more than sufficient for the GAIA interferometer system. However, there is no indication that this value is an upper limit for HCB bonding strengths. In silica bonding strengths of more than 50 MPa has been shown, which is the strength of silica.⁸ This should be achievable for HCB SiC–SiC bonds.

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