

Demonstration and challenges for joining technology based on direct bonding usable for construction of (large) structures in space

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Abstract A direct bonding process relying on van der Waals forces offers an ideal combination of easiness to assemble and material compatibility. Such a bonding procedure, also denoted as optical bonding, is already known and used frequently in different applications. However, there are strict requirements to achieve optical bonding: (1) a high level of cleanliness of the surfaces and (2) low roughness (RMS roughness <2 nm and preferably <0.5 nm). The first condition will be possible to realize in space, since the vacuum prevents the absorption of a thin water layer present on all surfaces under atmospheric conditions and forces a desorption of all residual water. Also, particle contamination will be minimized, due to the absence of strong capillary forces which attract particles if present and the absence of particles in space, providing a suitable and without residue removable protection prior to bonding. The second condition is now state of the art of silicon carbide (SiC) polishing and can hence be realized. While meeting the requirements, theoretically very large adhesion forces can be realized in vacuum. Surfaces have been polished according to the requirements. The forces measured on these surfaces are nearly as high as theoretically predicted and demonstrate the proof of principle of direct bonding of

SiC under ambient conditions and in vacuum. However, the realization of the required flatness over large contact areas is still a challenge. Furthermore, since the surfaces display a really low roughness, extremely clean handling and bonding conditions need to be realized to avoid the spontaneous adhesion of small particles which would as such prevent direct bonding of larger areas.

Keywords Silicon carbide · Direct bonding · Adhesion · Construction in space

1 Introduction

The main goal of this study is the development of self-joining technology of silicon carbide, based on spontaneous adhesion of super-smooth surfaces.

Future space missions such as space-based astrophysics observatory, permanent space stations or a spaceport in lower earth orbit (LEO)/lunar orbit which are currently under consideration need stiff structures so large that the limitations by the size of the available spacecraft (Ariane envelope is 4.5 m diameter \times 10 m length) have to be overcome by either using deployable structures or in-orbit assembly technologies. Deployable structures comprising low-density structural elements have been explored in the past and even in an in-orbit demonstration assembly using the Shuttle [1, 2]. However, they did not always show the ability to guarantee high eigenfrequencies together with precision positioning after deployment and a high stiffness combined with very low thermal expansion. Especially, astrophysics missions require increased apertures and focal length, which mean larger dimensions coupled with a mirror surface accuracy in the order of nanometers and platform stabilities in the range of a few microns.

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Among all possible materials to be used, glass and carbon fiber-reinforced composites show the best elastic modulus to design ratios. However, composites show at long durations and high and low temperatures, thermal aging and degradation, especially on direct exposure to the sun [3]. Silicon carbide (SiC) is a much stiffer and durable material, currently used for precise instrumentation of frames and mirrors. However, joining of SiC elements is not an easy task. A direct bonding process relying on van der Waals forces would offer an ideal combination of easiness to assemble and material compatibility. In literature, these forces are also denoted as optical bonding. To achieve optical bonding, two main conditions have to be met [4]: (1) high level of cleanliness of the surfaces and (2) low roughness, together with a high flatness. The first condition will be easily realized in space, since the high vacuum present in space prevents the absorption of a thin water layer present on all surfaces under atmospheric conditions. Also particle contamination will be minimized, due to the absence of strong capillary forces which attract particles if present and the absence of particles in space. The required low roughness is now state of the art of SiC polishing for optical applications and can hence be realized. Direct bonding is already used for the combination of SiC wafers in ground applications; however, here typically a rather stringent control of humidity and contamination is required to ensure a successful process. Additionally, often a subsequent heat step is part of the bonding process which stimulates the bonding on the atomic level due to higher mobility and total removal of water residues.

The surfaces also need to be conformal ensuring precise positioning of the joint; however in most cases, the contact may be made in a flat-on-flat configuration requiring precise positioning, since corrections after the bonding (re-positioning or re-alignment) are not possible once the surfaces are in contact. To meet such very demanding requirements, additional positioning tools like embossed positioning areas may be considered. The adjoining surfaces will be embossed with a higher roughness to ensure self-alignment of the joints.

A big challenge will be the realization of flatness levels of less than 5 nm for areas of several cm² may be required; currently, only a flatness level of 20 nm has been achieved. The parameters of influence for direct bonding/spontaneous adhesion are explored and known [5] as well as calculations of the van der Waals forces between solid surfaces at the nanoscale [6]. In the study of Kudryavtsev et al. [6], the influence of roughness, and to be more particular the height distribution of the asperities, their curvature and density is highlighted to understand the adhesion forces and to develop strategies to prevent unwanted adhesion. State-of-the art bonding of SiC uses liquid bonding agents and/or high temperatures and is as such only with

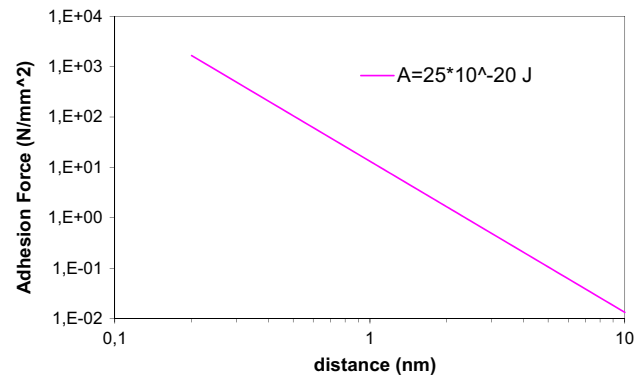


Fig. 1 Computed adhesion force in N/mm² for different achievable distances of two parallel SiC plates

difficulties applicable in space. Therefore, it is one of the major show stoppers so far for in-orbit assembly of structures constructed from SiC larger than the envelop provided by spacecraft. Joint concepts should provide rigidity and capability to transmit axial and transverse loads, ease of assembly, and simplicity of the joint design and the minimization of joining mechanisms. The joint system must have especially a good thermal compatibility with the low thermal expansion characteristics of the structural elements. Lastly, the weight of a particular joint concept is, of course, a significant consideration. In contrast to the classical ball/socket and probe/drogue as developed by Grumman or to welded joints [7, 8], a direct bonding process relying on van der Waals forces would offer an ideal combination of easiness to assemble and material compatibility.

The target application is the joining of SiC beams via direct bonding in space for the construction of large stiff frames for exploratory experiments and missions (optical quality).

The theoretical bond strength (van der Waals) forces, which can be developed between two parallel plates in vacuum, can easily be computed while knowing the Hamaker constant and using the following equation [9]:

$$F_{PP} = \frac{A}{6\pi d^3}, \quad (1)$$

with A as Hamaker constant and d as distance between the parallel plates.

The Hamaker constant of SiC in vacuum is known and is about $24\text{--}26 \times 10^{-20}$ J [9].

The expected adhesion strength is thus dependent on the surface quality (flatness and roughness) which limits the possible distance of contact of two parallel plates (see Fig. 1).

To achieve stable direct bonding, surface qualities as specified above are required: the rms roughness must be <2 nm and preferably even <0.5 nm; the surface flatness

should be ~ 5 nm, and areas for bonding need to be at least $1\text{--}10\text{ cm}^2$. The Young's modulus of sintered SiC is around 400 GPa. If bonding of chemical vapor deposition (CVD)-coated components is successful, lightweight constructions compared to current structures are possible since the Young's modulus of CVD SiC is 490 GPa due to the higher density (10–20 % weight reduction). The target bond strength is >0.1 MPa, which is a fraction of the theoretical possible strength for a distance of 2 nm; this value is about 1.6 MPa for 1 cm^2 bonding surface (Fig. 1).

2 Materials and methods

Different suitable flat samples with dimensions of $20 \times 20\text{ mm}^2$ and with a diameter of 12.7 mm were produced by Xycarb B.V. using CVD technology developed by Xycarb. The latter samples were specially doped throughout the whole material with nitrogen to increase the electrical conductivity ensuring a maximum possible adhesion force and discharging of electrostatic currents. The samples were further polished to achieve maximum flatness and minimum roughness (see Figs. 2, 3).

The macroscopic convexity/surface flatness was sufficient for these areas, which is equivalent to the area used for the determination of the adhesion/bonding forces (approx. 0.5 cm^2).

Also, for comparison, a piece of sintered and polished SiC, as supplied by Kyocera Nederland, was incorporated in the sample selection; the characteristics are listed in Table 1.

However, to test also substrates with a minimum of roughness, commercially available and CMP single-side polished N-doped SiC (4H-N) wafers were acquired from University Wafer, USA, and used in this study as well. Chemical mechanical polishing/planarization (CMP) is

a process of smoothing surfaces with the combination of chemical and mechanical forces. It can be thought of as a hybrid of chemical etching and free abrasive polishing. The process uses an abrasive and corrosive chemical slurry (commonly a colloid) in conjunction with a polishing pad and retaining ring, typically of a greater diameter than the wafer. The pad and wafer are pressed together by a dynamic polishing head and held in place by a polymer retaining ring. The dynamic polishing head is rotated with different axes of rotation (i.e., not concentric). This removes material and tends to even out any irregular topography, making the wafer flat or planar as well as smooth.

The surface morphology of the acquired wafer was measured and evaluated using AFM and a relevant picture is shown in Fig. 4.

Clearly, a decrease in surface roughness leads to an enhancement of stiction potential, as seen in Fig. 1. Here, and not initiated, contamination particles adhere quickly and spontaneously to a free surface.

Additionally, control experiments using an Si wafer with a surface roughness of 0.3 nm were used to test the cleanliness of the experimental setup.

Table 1 lists all samples with their characteristics used in this study.

3 Results

3.1 Test of direct bonding and mechanical testing

Direct bonding experiments of the polished surfaces were performed, together with a mechanical testing/characterization of the achieved bonding strength. For the testing of occurrence of spontaneous adhesion/stiction and for the quantitative determination of the adhesion force, the Universal NANO-mechanical Tester (UNAT) from ASMEC

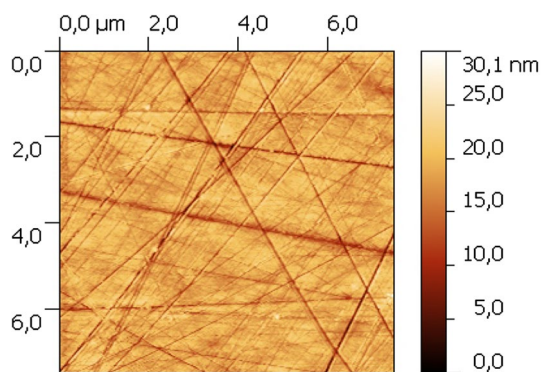


Fig. 2 Surface topology as measured by atomic force microscopy (AFM) of CVD-SiC as manufactured by Xycarb and mechanically polished, rms = 3.2 nm

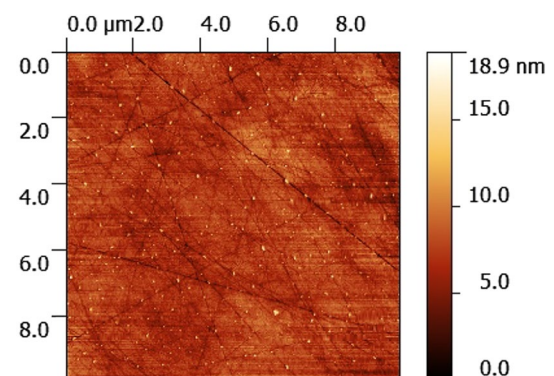


Fig. 3 Surface topology as measured by AFM of CVD-SiC doped with N as manufactured by Xycarb and mechanically polished, rms = 1.8 nm

GmbH, Radeberg, Germany, was employed [12]. The UNAT is an extension of the established nanoindentation technique. In contrast to most nanoindenters available in the market, the measuring head works in compression

Table 1 Samples used for adhesion testing

Sample and origin	Area	Roughness, RMS (nm)
Si wafer	Approx. 400 mm ²	0.3
SiC polished, Xycarb	Approx. 525 mm ²	3.2
N-doped SiC polished, Xycarb	Diameter 12.7 mm	1.8
N-doped SiC wafer, University Wafer	Diameter 50.8 mm	0.2
SiC sintered and polished, Kyocera	Approximately 1,600 mm ²	3.3

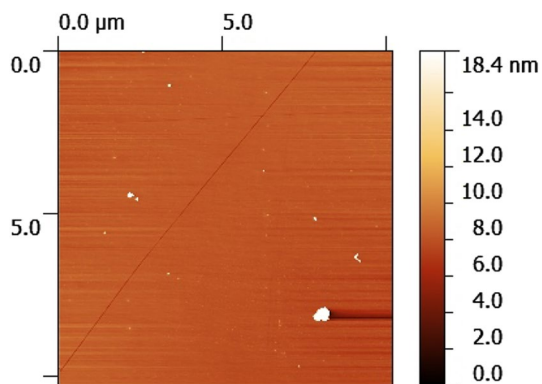


Fig. 4 Surface topology of the SiC N-doped wafer as purchased from University Wafers, rms = 0.2 nm

Table 2 Specifications of UNAT tests

Specification	Value
Maximum normal and lateral force	±2,000 mN
Digital force resolution	≤0.1 μN
Noise-level force measurement	≤6 μN
Digital displacement resolution	≤0.01 nm
Noise-level displacement measurement	≤1 nm

Fig. 5 Picture of the test tool showing the Si-IR lens on top glued to the adapter for the UNAT tester and 3D plot of the interferometric determination of the surface topology of the used Si-IR lens



mode as well as in tension mode, so that micro tensile and adhesion tests are possible. The conventional diamond tips can be replaced by tips of any shape and material without loss of resolution. Thus, the actual material couples of an application may be reproduced and modeled in the laboratory. The instrument can work under load control as well as displacement control in “open loop mode” (only the maximum force or displacement is controlled) or “closed loop mode” (every point of a curve is controlled). The maximum data rate is 64 points per second, so that very fast measurements are possible. The main instrument specifications relevant for an adhesion testing experiment are listed in Table 2.

For the adhesion testing experiments, either a silicon infrared lens with a surface roughness rms = 2 nm and a curvature radius of 123 mm (Fig. 5; Table 3) or a special Xycarb-manufactured SiC lens with a curvature radius of 19.4 mm and a surface roughness of 1.8 nm (Fig. 6; Table 3) was used as counter surface brought in contact with the polished SiC samples.

Table 3 shows an overview of all counter test bodies with their characteristics used in this study.

For the adhesion experiments, displacement control with different displacements resulting in normal contact forces ranging from 2 to 800 mN was applied (Fig. 7).

The adhesion measurements were performed at controlled humidity conditions (26 % RH). Typically, in a sufficiently clean experimental setup, already snap-on or spontaneous adhesion during the approach and surface position finding procedure could be observed (Fig. 8).

Subsequently, the measurement sequence was run and different values for the displacement in contact which could be translated into different normal forces were applied. Figure 9 shows a plot comparing the measured forces for an Si wafer and for the SiC wafer in contact with the Si-IR lens.

Subsequently, the same experiment was performed using the specially manufactured SiC lens. Again, snap-on onto all tested samples could be observed as well as the adhesion forces measured (Fig. 10).

The results of the adhesion measurements are also shown in Table 4 and Fig. 11. Again, the smaller value of

the adhesion forces measured with the SiC lens is due to the smaller radius of the SiC lens compared to the Si-IR lens and hence a resulting smaller contact area. In Table 4, the contact pressure is calculated as well, using Hertz' equations as a first approximation for the contact radius and pressure. In this approximation, it is assumed that the adhesion force acts as the normal force in the usual Hertzian approximation. A second note that is to be made when using this approximation is that due to the large diameter of the test bodies, also asperities outside the Hertzian contact zone will make contact; see e.g., [14]. The average pressure will therefore be lower, while the effective contact radius will be larger.

Clearly, the adhesion forces determined for the contact SiC–SiC are in the range of the earlier measured values of adhesion forces for the systems Si–Si and Si–SiC and hence in the expected range [10, 11]. Consequently, direct bonding between well-prepared and smooth SiC surfaces is possible for the attempted purpose, namely to join constructions without the necessity of using glue.

In Fig. 12 the average contact pressure due to the adhesion is plotted for the different tests.

The adhesion value for the Si wafer is no longer the highest value due to the difference in elasticity between silicon and SiC. Comparing the values determined for the different SiC samples including the N-doped SiC, the adhesion contact pressure scales with the smoothness of the contact surface. The differences measured while using the Si-IR lens are almost negligible; obviously here the properties of the Si are dominant, with Si being the more elastic contacting surface.

Table 3 Counter test bodies used for adhesion testing

Sample and origin	Area, curvature and roughness
IR-Si lens, Edmund Optics	Diameter 12.7 mm, radius = 123 mm, rms = 2 nm
Polished SiC lens, Xycarb	Diameter 4 mm, radius = 19.4 mm, rms = 1.8 nm

Fig. 6 Picture of the test tool showing the polished manufactured SiC lens on top of the test tool and 3D plot of the interferometric determination of the surface topology of the used SiC lens



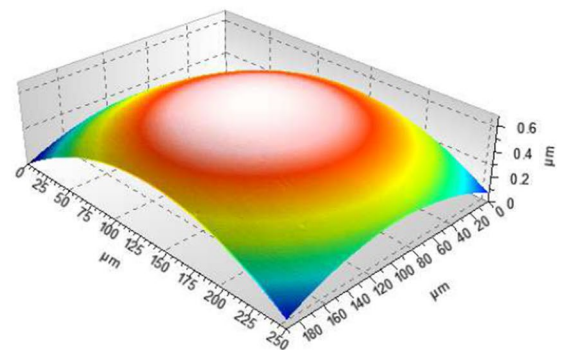
The adhesion contact pressures with the SiC lens are a factor 2.5–3 higher than the measurements using the Si-IR lens. For the difference between the measurements using the Si-IR lens and the SiC lens in contact with the same material, two possible causes are identified. The SiC lens has a relatively small contact radius; therefore the number of asperities touching outside the Hertzian contact area is relatively large. The α_s value as defined in [14] is therefore larger for the SiC lens, indicating that it is further off the Hertzian theory, which is used to calculate the adhesion contact pressure in Fig. 12. Thus, the effective nominal contact area is larger. Since the roughness of both Si-IR lens and SiC lens is about equal, this indicates also a larger real relative contact area.

The second factor is the difference is surface chemistry, which can cause either of the surfaces to be more hydrophilic. This factor also depends on the degree of cleanliness of the surfaces.

3.2 Test of proof of concept by measurement of adhesion in vacuum

Alternatively to the originally planned testing of the adhesion in vacuum by AFM, a second option has been employed for testing of the proof of concept of direct bonding of SiC as joining technology in space. For this, a new and specially designed variant of the UNAT for testing in a scanning electron microscope vacuum environment was used [13]. The experiments were performed at the laboratory of Experimental Methods in Material Science (Prof. C. Motz), University of the Saarland, Germany.

The experiments employed the same test specimen (SiC lens-Xycarb) as already used for the experiments and measurements under ambient conditions; the experimental procedure followed the earlier described protocol. As counter surface, only the SiC wafer was selected since the measurement of adhesion forces is very difficult and requires some experience, especially for the cleaning of the surfaces prior to the experiments. The cleaning protocol used at the University of Saarland differed slightly from



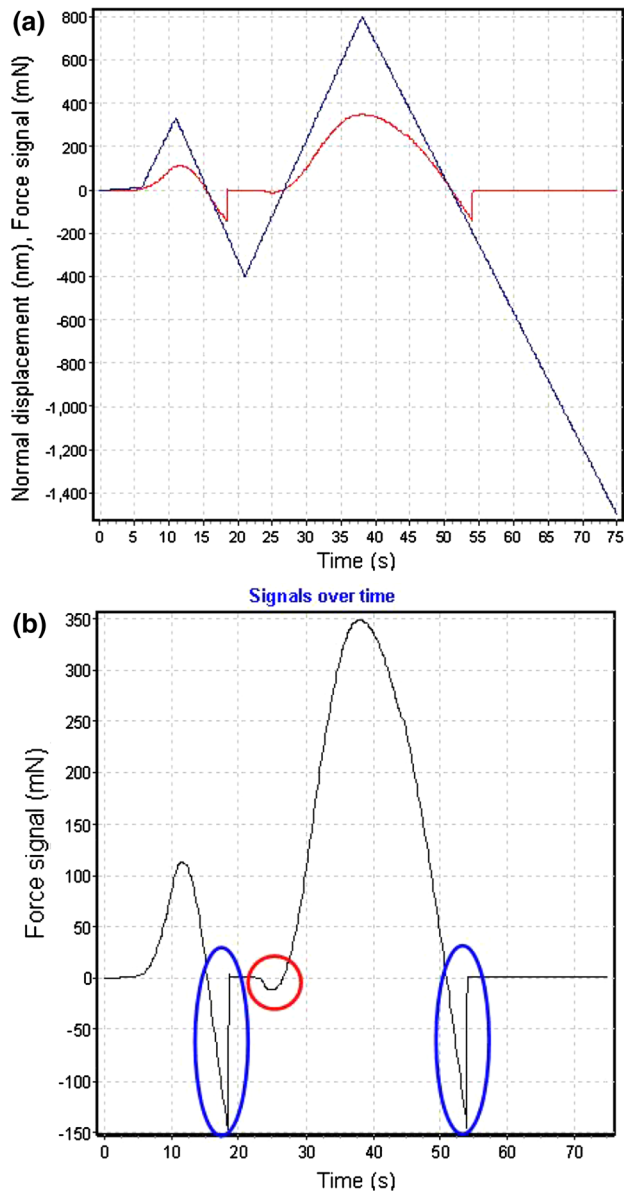


Fig. 7 **a** Measurement sequence used in the UNAT experiments (displacement controlled—*blue*) and obtained normal force signal (*red*) during the experiment over time. The measurement tool is firstly brought into contact with the counter surface (positive displacement) and then retrieved (negative displacement). Subsequently, a second cycle was executed, **b** zoom in of the obtained force signal in mN over time showing clearly pull-off (stiction during retrieval of the lens—*blue*) and snap-on (*red*) during approach

the procedure used at TNO (dry vs wet-dry). This may result in a different level of contamination which directly influences (degrades) the measurable adhesion forces. Therefore, the values obtained under ambient conditions are somewhat smaller (approximately, 5 mN), than for the measurements under ambient conditions (11 mN). However, the envisaged measurements in vacuum could be done

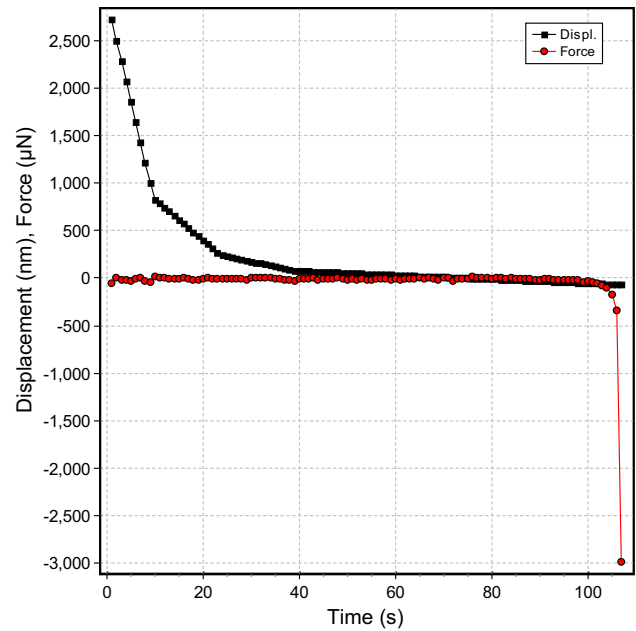


Fig. 8 Displacement and force signal during approach of the SiC wafer by the SiC lens. The approach is displacement controlled and occurred using different approaching speeds dependent on the expected distance. The force signal recorded shows strong attraction while getting closer to the surface at about 100 s experimental time (spontaneous adhesion)

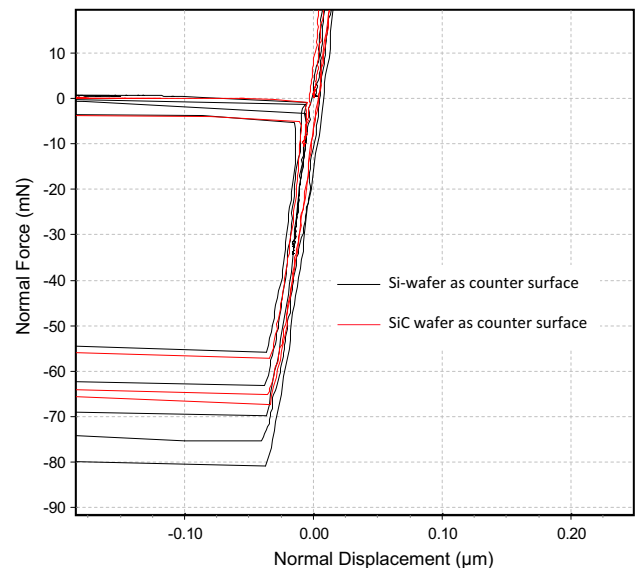


Fig. 9 Force–displacement graph obtained during testing of the silicon wafer (*black*) and of the SiC wafer (*red*) by the Si-IR lens using the UNAT. The silicon lens was pressed slightly against the surfaces to assure full contact. Then the lens was pulled up. The required (negative) force is a measure for the amount of adhesion. The Si wafer shows an adhesion force of approximately 80 mN and the SiC wafer of approximately 65 mN. Clearly the snap-on during approach can be observed repeatedly

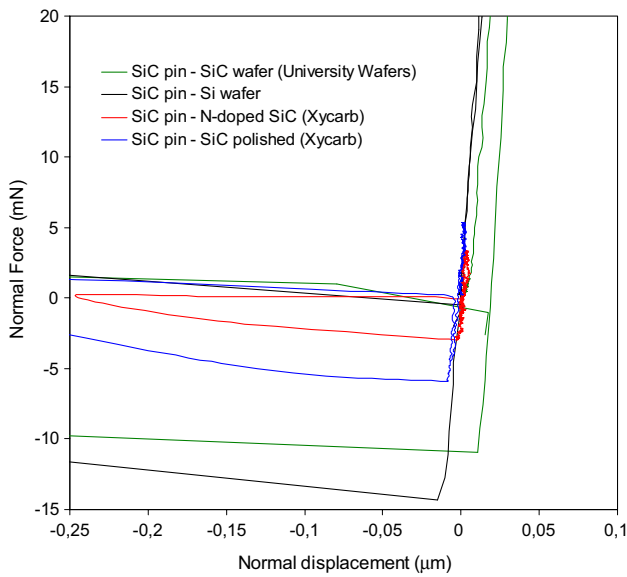


Fig. 10 Force–displacement graph obtained during testing of the silicon wafer (black), the SiC wafer (green), the polished N-doped SiC sample (Xycarb, red) and the polished SiC sample (Xycarb, blue) by the SiC lens using the UNAT. The measured maximum adhesion values for the Si wafer are approximately 14 mN, for the SiC wafer approximately 11 mN for the N-doped polished SiC sample ca 6 mN and for the polished SiC sample approximately 3 mN

(see Fig. 12). The measured adhesion value for testing in vacuum is approximately 3.5 mN (Fig. 13).

4 Discussion of the results

The measurements show convincingly that spontaneous adhesion between SiC surfaces of sufficient quality is feasible and measurable. A quick calculation of the expected adhesion forces between a flat surface and a sphere (measurement setup) even under ambient conditions should lead to substantial and measurable forces (Fig. 14).

Again, the theoretical adhesion force can be calculated using the DMT (Derjaguin, Muller, and Toporov) theory

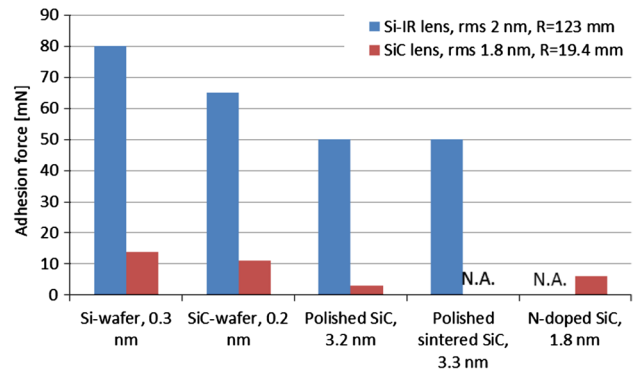


Fig. 11 Plot of the measured adhesion forces for the different combinations of samples and test bodies

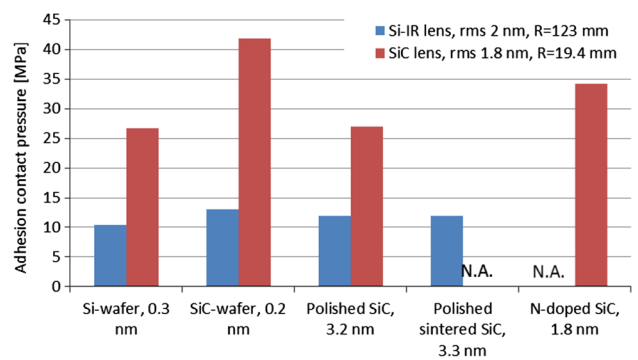


Fig. 12 Plot of the measured adhesion contact pressure for the different combinations of samples and test bodies

and while knowing the Hamaker constant for the interacting bodies, in this case assuming a water lens layer on the surfaces (11×10^{-20} J) [9]. In this case, the equation for a plate–sphere contact can be employed:

$$F_{PSphere} = \frac{A \cdot R_{Sphere}}{6\pi d^2} \tag{2}$$

For the computation of the theoretically achievable adhesion forces, the influence of capillary forces is neglected. The measured data are somewhat larger than the theoretical

Table 4 Tested material combinations and resulting adhesion forces and contact pressure due to adhesion

Test tool, rms (nm) and radius	Tested surface and rms (nm)	Measured $F_{adhesion}$ (mN)	Contact pressure (MPa)	Contact radius (μ m)
Si-IR lens, 2, $R = 123$ mm	Si wafer, 0.3	80	10.4	49
Si-IR lens, 2, $R = 123$ mm	Polished SiC, 3.2	50	11.9	36
Si-IR lens, 2, $R = 123$ mm	Polished sintered SiC, 3.3	50	11.9	36
Si-IR lens, 2, $R = 123$ mm	SiC wafer, 0.2	65	13.0	40
SiC lens, 1.8, $R = 19.4$ mm	Si wafer, 0.3	14	26.7	12.9
SiC lens, 1.8, $R = 19.4$ mm	SiC wafer, 0.2	11	41.8	9.1
SiC lens, 1.8, $R = 19.4$ mm	N-doped SiC, 1.8	6	34.2	7.5
SiC lens, 1.8, $R = 19.4$ mm	Polished SiC, 3.2	3	27	5.9

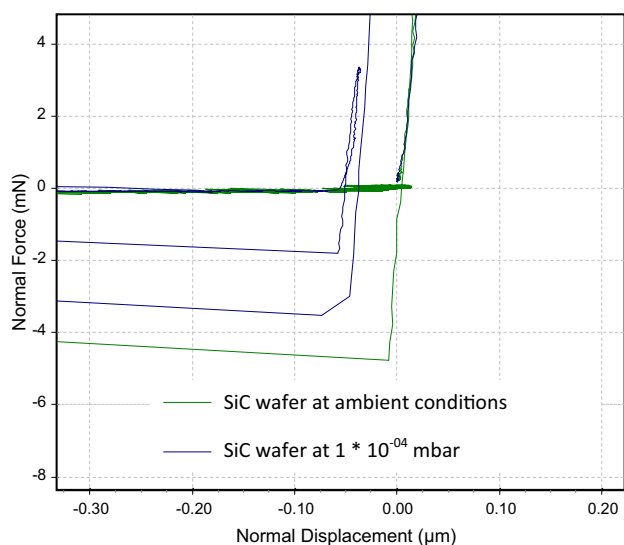


Fig. 13 Force–displacement graph obtained during testing of the silicon carbide wafer under ambient conditions (green) and at 1 e^{-04} mbar (blue) by the SiC lens using the UNAT SEM2. The measured adhesion values are for the SiC wafer approximately 5 mN for testing under ambient conditions and approximately 3.5 mN for testing in vacuum

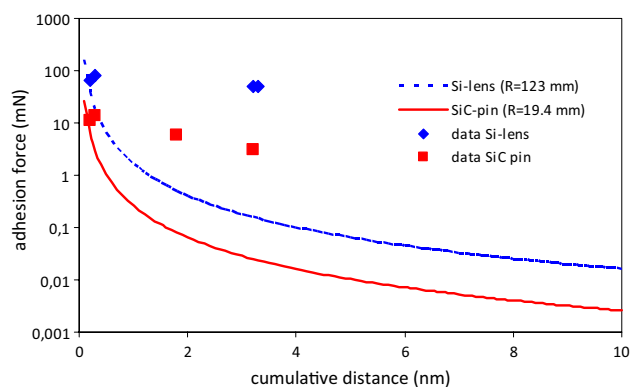


Fig. 14 Computed adhesion force in mN for different achievable distances using the two available spherical counter bodies

expectations. This is not surprising, since there is a residual roughness of the real surfaces enabling capillary condensation of water and resulting in larger real contact areas than expressed by the Hamaker equation, assuming perfect curvature and smoothness of the counter surfaces. In case of a good contact (very small contact distance), the area of the meniscus at the edge of the contact area will be rather small and the contribution of the capillary forces indeed irrelevant.

However, the realization of the required flatness over large contact areas is still a challenge, as stated before. Furthermore, since the surfaces display a really low roughness, extremely clean handling and bonding conditions need to be realized to avoid the spontaneous adhesion of small particles which would as such prevent direct bonding of larger areas.

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