Silicon-Carbide-Bonded Diamond Components for Harsh Environments – Cost-Effective Components with Outstanding Properties

Due to their excellent wear and corrosion resistance, advanced ceramics may become enablers for subsea systems. The Fraunhofer AdvanCer Alliance has started the internal Fraunhofer project CS³ – Ceramic Subsea Systems – addressing the designing, reliability, manufacturing and testing of key components for subsea applications. Cost-effective wear-resistant components based on SiC-bonded diamond materials were developed within the project.

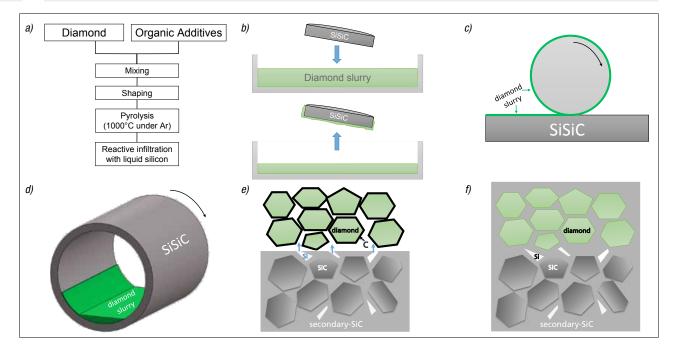


Fig. 1 a–f

Schematic view of the preparation of SiC-bonded diamond materials (after [9]): a) process flow, b-d) application of the diamond surface layer, e-f) infiltration and reaction bonding processes

Introduction

The availability of raw materials and new sources of energy are the major challenges for all industrialized countries. The

Keywords

SiĈ-bonded diamond wear parts, Electrical Discharge Machining (EDM), subsea systems exploitation of subsea reservoirs plays an increasingly important role. Due to their excellent wear and corrosion resistance, advanced ceramics may become enablers for subsea systems. New generations of pump systems, using silicon carbide SiCbonded diamond wear parts, extremely large zirconia separating cans and presM. Herrmann, B. Matthey, S. Kunze, M. Zins Fraunhofer Institute for Ceramic Technologies and Systems, Germany

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sure-resistant current feed throughs will become part of deep sea manufacturing systems. Successful business is often limited as there are reservations about the reliability of ceramics. The Fraunhofer AdvanCer Alliance will try to overcome these doubts by presenting new materials as well as the know-how for designing, manufacturing and testing key components for subsea applications. Within the internal Fraunhofer project CS³ – Ceramic Subsea Systems - the three Institutes Fraunhofer IKTS. Fraunhofer IWM and Fraunhofer IPK started to develop components based on SiC-bonded diamond materials for subsea applications. In the course of the project. complex wear parts and prototypes will be produced and tested in cooperation with industrial partners. The know-how in design and ceramic-specific calculation, manufacturing, joining and non-destructive testing will be available for interested partners.

Preparation and microstructure

Low-cost, superhard, wear-resistant materials are interesting for a wide range of wear applications with extended life time. As typical diamond-based wearresistant materials are produced by using high pressures, they have restrictions in dimensions and costs. Manufacturing of SiC-bonded diamond materials, by contrast, represents a promising route to obtain low-cost, superhard, wear-resistant materials since SiC-bonded diamond composites can be produced through reactive infiltration of diamond preforms with silicon [1-9]. This is a conventional ceramic technology analogous to those used to manufacture SiSiC components (Fig. 1 a). First, diamonds are mixed with the corresponding binders and solvents and granulated. Afterwards they are compacted by pressing or other shaping technologies.

After pyrolysis in argon, the porous green body consisting of diamond and the carbon derived from the binder is infiltrated with liquid silicon at 1450–1600 °C. The process is schematically shown in Fig. 1 e–f. During infiltration, liquid silicon reacts with carbon from the binder and partially with the diamond grains (Fig. 1 e–f). The reaction of diamond with infiltrated silicon to SiC is accompanied by an increase in volume of 266 % (129 % for reaction with graphite). For that reason, there are

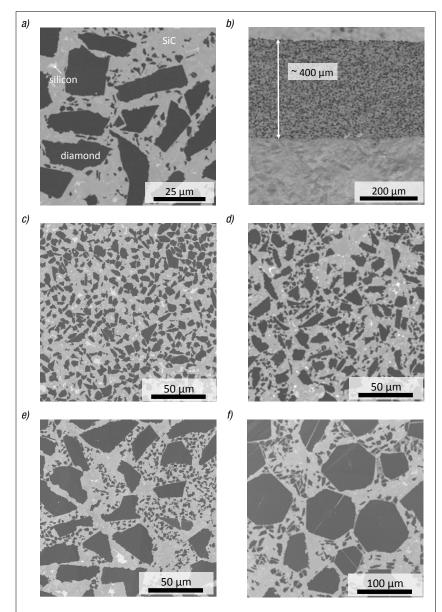


Fig. 2 a–f

Microstructure of SiC-bonded diamond materials: a) micrograph showing the different phases, b) interface between SiC-bonded diamond and the SiSiC substrate, c)–f) micrographs giving an idea for the possible variations of the microstructures

limitations in terms of packing density and minimum pore channel diameter in the diamond preform. However, the infiltration works effectively for diamond grain sizes larger than $10 \,\mu$ m. Fig. 2 a shows the resultant SiC-diamond microstructure. It consists of diamond grains embedded in a three-dimensional SiC framework. Unreacted, residual silicon is found in the interstices between some of the SiC grains. The SiC grains grow with special orien-

tations on the surface of the diamonds. This is an indication of the direct chemical bonding between diamond and SiC [6]. This microstructural feature is the reason for the outstanding properties of the material. In dependence of the used diamond powder, completely different microstructures (Fig. 2 c–f) and, therefore, properties can be achieved. Diamond contents of up to 59 vol.-% and silicon contents less than 5 vol.-% Si have been achieved up to now. As mentioned before, the preparation process is very similar to the preparation of Si infiltrated SiC. Therefore, it is possible to produce components having the SiC-bonded diamond surface layer only in these areas, where wear resistance

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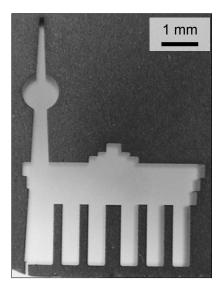


Fig. 3 Berlin landmarks machined in SiC-bonded diamond by wire EDM

Tab. 1 Characteristics of the SiC-bonded diamond materials

is required. This can be realized by cold isostatic pressing of SiC and diamond granulates as well as by dipping and casting methods. The casting and dipping methods developed within the project (Fig. 1 b–d) are very effective for the preparation of complex shaped wear-resistant components.

As schematically shown in Fig. 1 e, Si-SiC can be used as substrate for dipping or casting with a diamond slurry. Typical thickness values of the SiC-bonded diamond surface layer prepared by these methods are between $300-500 \mu$ m. However, this process can be repeated several times resulting in higher thickness values. Fig. 2 b shows the cross-section of a SiC-bonded diamond layer on SiSiC. No defects or delaminations were observed at the interfaces. Additionally, SiC-bonded

diamond materials can also be produced as bulk components by traditional casting or tape casting technologies.

As the components can be green machined after pyrolysis and the volume change is nearly zero during Si-infiltration, SiC-bonded diamond components can be produced near netshape and, thus, very cost-efficiently.

Due to the high wear resistance of the SiC-bonded diamond materials, final machining by cutting and grinding is limited. Lapping and polishing of the surfaces is possible but time consuming. Nevertheless, laser cutting and Electrical Discharge Machining (EDM) are attractive alternatives for machining the SiC-bonded diamond materials. The general advantage of EDM is its applicability independent of the mechanical properties such as strength,

Material	Diamond Size	Diamond Content	Free Si	KIC	Strength B3B	Hardness HK2	Thermal Conductivity
	[µm]	[vol%]	[vol%]	[MPa•m ^{1/2}]	[MPa]	[GPa]	[W/m·K]
D10	10	45	5	$\textbf{4,8} \pm \textbf{0,4}$	480 ± 40	$48,6\pm1,6$	260 ± 5
B50	50	43	3	$4,9\pm0,3$	505 ± 40		400 ± 5
T100	100	57	1				500 ± 5

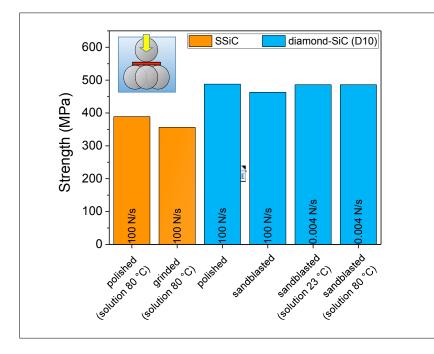


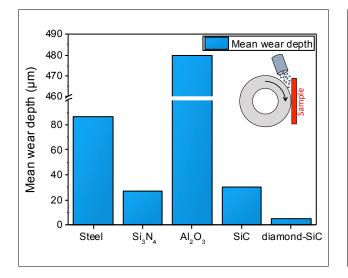
Fig. 4

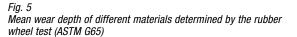
Strength of the SiC-bonded diamond material with polished and sandblasted surfaces in air and in salt solution at room temperature and 80 °C in comparison to a commercial SSiC-material

hardness, wear resistance or toughness. The only requirement is that the materials need to have an electrical conductivity $\kappa > 0.01$ S/cm. Therefore, the electrical conductivity of the SiC-bonded diamond material had to be adapted. So, materials with the conductivity of $\kappa \ge 2,50$ S/cm could be machined by wire or die sinking EDM. The samples were machined by wire EDM on an industrial fine-wire EDM machine of the type AC Vertex 1F. As shown in Fig. 3, a contour was cut out in the shape of the Berlin TV Tower and the Brandenburger Tor with a circumferential length of 40 mm after a processing time of 60 min.

Properties

An overview of the properties obtained for the SiC-bonded diamond materials is given in Tab. 1. The mechanical properties were measured by means of a ball-on-3balls test using ceramic balls (Ø 23,8 mm) and circular ceramic specimen disks





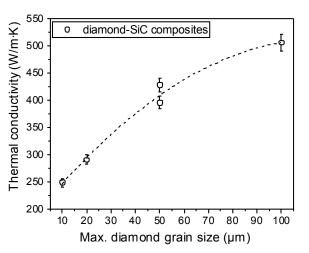


Fig. 6 Dependence of the thermal conductivity on the diamond grain size (after [8])

(Ø 30 mm, thickness 2 mm). The mechanical strength of the material was determined on polished and as sintered and sandblasted surfaces. Additionally, measurements in salt solutions at room and elevated temperatures and low strain rates were carried out to evaluate effects of the SiC-bonded diamond composites.

Fig. 4 shows that the SiC-bonded diamond materials reached strength values between 450-500 MPa, and neither a bad surface finish, like sandblasting, nor the water environment had a negative effect on the mechanical strength. The values are higher than the values of a standard SSiC material measured under the same conditions (Fig. 4). The materials show fracture toughness values between 4,5–5 MPa·m^{1/2}. These relatively high values are not caused by pull out of the diamond grains, which was not observed at fracture surfaces at all, but more likely by fluctuating internal stresses [8]. The diamond grain size - at least in the range between 10-50 µm - has also no effect on the strength values [6-8].

A comparative evaluation of the abrasion resistance of the SiC-bonded diamond materials was made using abrasion tests according to ASTM G65, in which a steel wheel was used in combination with aqueous sand suspension to induce higher abrasive effects. The results (Fig. 5) show that the abrasion resistance of the SiCbonded diamond composite is even much higher than that of other structural ceramics like silicon nitride or silicon carbide. Surface analyses of the SiC-bonded diamond material after the tests revealed that only a part of the silicon carbide phase was worn off and the diamond grains hardly showed any traces of wear. Similar high wear resistance was observed during sandblasting. The wear resistance was 10 times better than the values observed for a dense commercial B_4C material [8]. Investigations of the tribological properties of these materials in oscillating sliding tests with steel and Si_3N_4 balls as mating bodies have demonstrated the great potential of this class of materials for tribocomponents. Although they contain only about 50 vol.-% of diamond, they exhibit a friction coefficient under dry running conditions as low as 0,1–0,2. These values and the wear resistance are very similar to polycrystalline diamond materials and

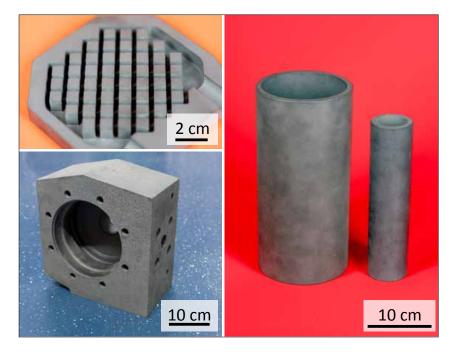


Fig. 7 Variety of SiC-bonded diamond components produced (heat sink, tubes and a casing of a radial flow pump (SICcast Mineralguß GmbH) with an inner SiC-bonded diamond surface layer)

CVD diamond films as the SiC matrix is slightly worn in the running-in period. After that, only the diamond is in contact with the counter body [8].

Investigation of the corrosion stability in hot acids showed a high stability. No reduction in strength was observed [7]. In hot bases, a partial dissolution of free Si existing in the material takes place. However, strength values of \geq 400 MPa can be maintained [7]. This is due to the high strength of the SiC-diamond network and the low content of free Si (\leq 5 vol.-%), which is substantially lower in comparison to SiSiC materials, and thus, the corrosion behaviour of the SiC-bonded diamond materials is more similar to the behaviour of SSiC than of SiSiC.

Besides the excellent wear and corrosion resistance the material exhibits also a very high thermal conductivity. The dependence of thermal conductivity as a function of the diamond grain size is shown in Fig. 6 [8]. Even with low diamond grain sizes thermal conductivity values of more than 250 W/m·K can be achieved. For the largest diamond grain sizes tested in the project more than 500 W/m·K could be measured. Fig. 7 shows some of the components produced from the SiC-bonded diamond materials as compact material or as SiSiC material with a SiC-bonded diamond surface layer. The examples give an idea about the possibilities in shape and size of components, which can be produced by the developed technology.

Outlook

The results demonstrate the potential of SiC-bonded diamond materials for the use under demanding tribological and wear conditions. They can be produced in different sizes and with complex shapes without implementing fundamentally new,

expensive technologies. Components with a wear-resistant SiC-bonded diamond surface layer only in highly stressed areas offer new possibilities for wear-resistant components with long lifetime e.g. tubes nozzles or inliners. The technology is developed in such a way, that it allows the production of prototypes of different shape and size in a reproducible way. At the moment in a Fraunhofer internal project the technology for the production of solid phase sintered SiC (SSiC) materials with a silicon carbide bonded diamond surface layer is under development. This allows a further improvement of the corrosion resistance of the components in comparison to materials with a SiSiC substrate.

Acknowledgement

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Literature

- Ekstrom, T.C.; Gordeev, S.K.: New carbide composites with extraordinary properties. Key Engin. Materials 161–163 (1999) 75–80
- 2 Gordeev, G.; et al.: US patent, US6, 2004 on Patent, 709,747
- 3 Herrmann, M.; Martin, H.P.: Verfahren zur Herstellung von Bauteilen mit einer Verschleißschutzbeschichtung, ein so hergestelltes Bauteil sowie dessen Verwendung I DE 10 2007063517 B3 (2009)
- 4 Herrmann, M.; Matthey, B.: Verfahren zur Herstellung von Verbundbauteilen und

ein mit dem Verfahren hergestelltes Verbundbauteil DE 10 2011 109573 (2011)

- Herrmann, M.; Matthey, B.; Schilm, J.: Bauteil hergestellt aus keramischem Werkstoff und Verfahren zu seiner Herstellung 10 2014 201 731.9
- 6 Matthey, B.; et al.: Microstructural investigation of diamond-SiC composites produced by pressureless silicon infiltration. Europ. Ceram. Soc. **37** (2017) 1917–192, http://dx.doi.org/10.1016/j.jeurceramsoc.2016.12.008
- 7 Herrmann, M.; et al.: Corrosion behaviour of silicon carbide–diamond composite materials in aqueous solutions. J. Europ. Ceram. Soc. **34** (2014) 2143–2151
- 8 Matthey, B.; et al.: SiC-bonded diamond materials produced by pressureless silicon infiltration. J. Materials research, DOI: https://doi.org/10.1557/ jmr.2017.218
- Herrmann, M.; et al.: SiC-diamond materials: Wear-resistant and versatile.
 cfi/Ber. DKG 91 (2014) E 39–E 43

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