Large-Sized, High-Performance Ceramic Components Made of Dense Silicon Carbide

In order to meet the increasing demand for large-sized and thick-walled components of dense SiC, specific material-related and technological developments have been carried out to enhance the feasibility limits. As a result, with optimized sintering technology, components of dense SiC could be realized with dimensions over the previous limits. There are, however, still limitations with respect to the maximum possible wall thickness of SiC parts which can be sintered to high densities. With this respect, modern hot-pressing technique with loading devices is a technical and economical alternative, provided that the parts to be produced are geometrically more simply shaped.

Motivation and objectives of developments

Since the realization of their sinterability in the 1980s, silicon carbide (SiC) ceramics have established in a wide range of technical applications due to their good mechanical properties from room temperature up to temperatures of 1500 °C in air, their outstanding thermal conductivity as well as their excellent chemical and abrasive resistance [1, 2]. This dense, sintered SiC-grade (S-SiC) provides an excellent corrosion resistance to almost all chemical substances of the entire pH range and a very favourable tribological behavior under a variety of conditions. These basic properties result mainly from the predominantly covalent chemical bonding in the SiC-compound, which, on the other hand, also leads to a high Young's modulus, high stiffness and brittleness. High stiffness, in combination with low thermal expansion and density, however, are also reasons for the use of SiC-parts for light-weight constructions like mirrors etc. in avionics and space applications [3]. Concerning brittleness, which remains as a kind of disadvantage of SiC-ceramics, usually can be managed by a careful handling, as broadly experienced in every days industrial practice.

Keywords
large-sized SiC-parts, sintering, hot-pressing, applications

As the covalent chemical bonding of SiC affords sintering temperatures well above 2000 °C and is associated with about 18 % shrinkage, the sizes of the manufactured SiC components were limited under technological and economic points of view as well as the yield of faultless products. Because of this, manufacturers persuaded the so-called SiSiC technological route to produce the desired components, an Si-infiltrated, reaction-sintered SiC-grade, which, on the one hand, even has a higher brittleness than dense, sintered SiC, and on the other hand, has a significantly lower chemical resistance due to the presence of about 10 vol.-% free Si in the material.

In the combination of material-related and technical improvements, in particular the availability of sinter-active SiC powders and premix-compositions with a high reproducibility with respect to the shrinkage, and the use of sintering apparatus according to the latest state of the art in terms of temperature uniformity at sintering temperatures above 2000 °C and atmospheric control, FCT expanded increasingly the limits of sinterable S-SiC components. Simultaneously, besides the advanced SPS/FAST technology for hardly sinterable materials, also the hot-pressing technique experiences a renaissance particularly for SiC components with a very high wall thickness, which are no more possible to get fully densified by the pressureless sintering technique [4, 5]. Newly developed hot-press equipment with increased maximum tool dimensions and compaction pressure as well as devices for automatic tool charging enable to expand the feasibility limits and cost-efficiency of the hot-pressing technique constantly. Thus, these advances in sintering and hot-pressing now allow a cost-effective manufacturing of large-sized, dense SiC components, as it was not imaginable a few years ago. This opens up a whole range of new tech-

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nical possibilities and applications, some of which will be described hereafter.

Large-sized, dense SiC components

In many technical and industrial areas there is an increasing demand for corrosion- and abrasion-resistant ceramic components, such as for preparation processes, conveyor techniques, processing of food-, drug- and pharmaceuticals etc. For new plants, there is a tendency to increasingly larger units which also require larger ceramic components. Focused on this, own developments were carried out to provide such large-sized, highly precise and complex shaped components made of dense SiC. With this respect, the following examples can be mentioned:

Processing technology: active and passive mill components

Applications like these nowadays are hardly imaginable without the use of active and passive ceramic components, including ceramic balls and beads, especially with regard to so-called high-energy mills, which enable size-reductions by milling down to less than 0,1 µm resp. 100 nm. Thus, a variety of ceramic materials and components are more or less established for such applications in the meantime [6]. With this respect, rotors with complex ball-agitators and -guides as well as stators and mill-liners with diameters up to 400 mm and lengths up to 900 mm (Fig. 1) are increasingly used in materials processing, like the high-energy mills mentioned [7]. The use of these ceramic components targets to avoid product contamination by abrasion and to increase mill power with respect to lifetime, throughput and achievable fineness of products. Mill-liners made of SiC provide the additional advantage of a high thermal conductivity, which allows very efficiently the dissipation of the abrasion-favouring frictional heat of the grinding process from the system. Besides the impressive dimensions of these parts, which are even about 20 % larger in the so-called "green", un-sintered state, they have already a weight of up to about 60 kg, which requires special techniques and devices for their handling and processing.

Well proved have also roll-sleeves for calendars for the treatment of contamination sensitive pastes for electronics, paints and coatings, cosmetics and nutrition but also of abrasive materials. These sleeves, which are geometrically relatively similar to the mill liners, were already produced in small series with dimensions up to 300 mm diameter and 900 mm length with a wall thickness of 15–20 mm. The finished status has a slightly convex crowning and finely polished outer surface and a honed cylindrical inner surface with shape- and position-tolerances in the micron range (Fig 1.).

Applications in chemical technology

In chemical technology, there are a variety of applications where dense SiC components were increasingly used advantageously due to their excellent corrosion and abrasion-resistance as well as high thermal durability and conductivity. Examples include reaction retorts, tubes for rotary tube furnaces and other customer-specific products which now can be produced with diameters up to 600 mm and lengths up to 1400 mm. Even larger units require the use of appropriate joining procedures.

Very common applications in chemical engineering are heat-exchangers made of steel or graphite, depending on the application conditions. In cases where the chemical resistance of these materials is no longer sufficient, appropriate equipment with SiC is an alternative, with the most well-established design as tube heat-exchanger with SiC tubes. Disadvantages of these apparatus designs, however, are the often necessary large dimensions and the functionality of the seals against the peripheral apparatus parts. These facts increasingly favour other designs such as block- and annular gap-geometries that allow a more compact and more effective technical solution [8].

In cooperation with an established manufacturer of heat exchangers, such components were developed, being a challenge with respect to "green"-moulding as well as sintering. Desired final dimensions are blocks of Ø 360 mm × 255 mm, which require cylindrical SiC green bodies of nearly 100 kg. The introduction of the drilled holes, which accumulate to about 100–150 m in length, depending on the number and diameter of the holes, calls for a very precise and careful deep hole drilling technique for the fragile green bodies. At the same time, the walls between the vertical product- and the horizontal service-channels shall be as low as possible (≤4 mm) to achieve a good thermal exchange efficiency. Examples of this successfully executed development are shown in Fig. 2. With the availability of the deep-hole drilling technique, also other geometrically comparable SiC components are now feasible.

Fig. 1
SiC mill-liner (D= 300 mm × 900 mm) and mill-rotor with internal ball guiding structures (D = 200 mm)

Fig. 2
SiC- (CorreSiC®) annular gap- and block-heat-exchanger components with diameters of 360 mm and a weight of about 40 kg (block)

Wear protection and armor applications

In the field of material-handling and -conveying like in pumps, in various industrial fields there appear conditions which even with armoured pump-wheels lead to enormous abrasive and corrosive wear and thus limit the lifetime of such components to e.g. less than 3 months. For such an application pump-impellers made of SiC have been developed even with a desired flow optimized blade geometry, which can be economically realized by applying the established 5-axis green-machining

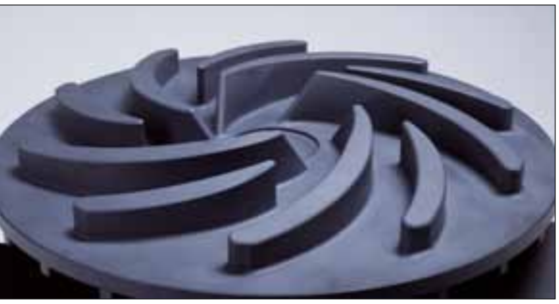

Fig. 3
SiC pump-impeller with front and backside blades, for applications under high abrasive and corrosive conditions, diameter 360 mm

Fig. 4
Large-sized SiC components for moulding thermoplastic materials with dimensions of about 530/420 mm × 400 mm, and a weight of about 27 kg

Fig. 5
Fully dense (≥3,19 g/cm³/≥99 % th.d.) HP-SiC block "as-pressed", with dimensions of 250 mm × 250 mm × 90 mm, and a weight of 18 kg

technology before sintering. An example of such a pump-wheel made of dense SiC with a diameter of 360 mm and front- and backside blades to reduce the axial bearing pressure is shown in Fig. 3. Preliminary

Fig. 6
Hot-press of type H-HP W 400 with multiple heating zones (Source: FCT Systeme GmbH)

application tests of such a pump-wheel have proved to be excellent so far and will markedly extend its running-duration and lifetime relative to pumping units with conventional wheels.

The limits of feasibility as dense, sintered SiC-parts were reached with components like the ones shown in Fig. 4 to be used for thermoplastic moulding of highly viscous materials like glass. Important for their use is a high temperature uniformity, which is promoted by the high heat conductivity of the SiC, and a reduced adhesion of the reshaped moulds. The production of these parts was also performed by isostatic pressing and green-machining of compacts of about 60 kg. The challenge for sintering these parts is characterized best by the fact that the green bodies of about 650/520 mm × 485 mm and a thickness of about 64 mm are subject of dimensional shrinkage in the range of 120 mm in its maximum direction. This must be managed by appropriate sintering conditions to avoid warpage or cracking. Due to the excellent controllability and good temperature homogeneity of the FCT sintering furnaces, these components could be sintered to densities of ≥97 % of the theoretical density. Meanwhile, first samples were shipped to the customer for technical evaluations.

If there are requirements for SiC parts with even greater wall thicknesses, limits were reached for their realization as a high-density component by sintering. An alternative in such cases is hot-pressing with appropriate apparatus, which was applied for e.g. the SiC blocks shown in Fig. 5 for ballistic protection devices. The implementation of the "pressure-assisted densification" for such large-volume parts required a newly conceived hot-pressing apparatus, equipped with multi heating zones and possibly an automatic batching device for increasing the throughput and thus the efficiency of the production of such blocks. The use of such a state-of-the-art hot-press enables up to eight hot-press cycles per day, meaning the production of up to 16 blocks per day for tandem pressings. Thus, from an economic point of view this hot-pressing technique is quite competitive with the sintering technique and provides an even better and more reliable material quality. This is due to the fact that by hot-pressing in contrast to sintering, an almost complete densification of SiC to ≥99 % th.d. is achieved. This provides an increase of the performance of such components, as their mechanical properties (among others) are exponentially dependent on the residual porosity. It seems obvious, that such optimized "HP-SiC" armor blocks with nearly complete density also offer an even higher ballistic performance.

Innovative sintering and hot-pressing technique

Resistance- or optional induction-heated hot-presses for hardly sinterable materials nowadays are available with press forces from 50 to 9000 kN and maximum operational temperatures of up to 2500 °C, suitable for components with maximum diameters up to 600 mm [4]. Unlike to pressureless sintering, however, by hot-pressing only components with a more simple geometry can be produced like discs, blocks or cylinders. In spite of this, they find increased use in particular for large-sized and thick-walled ceramic components, which cannot be sintered to full density any more. Especially the elimination of the final, residual porosity causes a further significant improvement in the material properties and the performance of such ceramic components.

Fig. 6 shows such a newly designed hot-pressing plant H-HP W 400, equipped with individually controlled 3 heating zones, which provide excellent temperature homogeneity within the hot zone. By means of up-to-date controller units, rate-controlled sintering-, respectively pressure-assisted densification cycles can be realized. Both measures enable to fabricate large-sized and thick ceramic components of high density and a very uniform microstructure. With such a new hot-press, the SiC blocks of 250 mm × 250 mm × 90 mm shown in Fig. 5 were compacted to ≥3,19 g/cm^3, meaning ≥99 % of the theoretical density.

Another new development is a batching device for hot-presses, allowing a more or less conti-process. With such a loading and unloading device as shown schematically in Fig. 7, up to 8 cycles for the mentioned SiC-blocks become possible per day. If the equipment even allows a multiple compaction of e.g. 2 parts per cycle, this results in 16 components in 24 h. It is obvious that such a conti-process provides a significant increase in throughput as well as the economy of the hot-pressing technique, in coincidence with best materials' properties.

The same is true for high-temperature sintering furnaces for SiC, which are also available with resistance- as well as induction-heating. They are suitable for many application areas as they can be operated with both vacuum and inert (partial pressure) atmosphere. A great advantage of these furnaces is that by means of a controlled gas supply and installed thermal afterburning, they allow debinding and/or pyrolysis of organic constituents and subsequent sintering of ceramic- or powder metallurgical components within one cycle, without transferring the kiln setting from one furnace to another one. Such furnaces, even with volumes of up to 6 m^3, reveal excellent temperature uniformity at the design temperature of 2500 °C, being a prerequisite for obtaining faultless, large-sized, dense SiC-components.

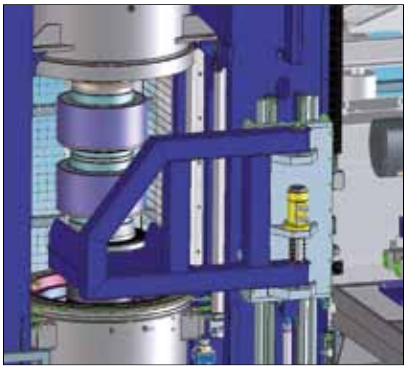

Fig. 7
Schematic representation of the loading- and unloading-device of conti hot-presses (FCT Systeme GmbH)

Summary and conclusion

By means of the described examples it is demonstrated, that the feasibility-limits of large-sized ceramic components made of dense SiC could be moved even further by purposeful technological developments and investments in the required equipment. With the availability of such large and high-performing ceramic components, new and innovative, more effective and/or more cost-efficient technical solutions and processes can be realized. It is important for users and customers that these parts do not have to be extremely expensive per se, but can be made in compliance with some boundary conditions and realistic demands concerning surface properties and geometrical tolerances to adequate and customers' acceptable costs and prices, particularly if the associated benefits of a longer life-time, lower maintenance and repair costs and higher overall performance are taken into account.

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References

[1] Wötting, G.; Simolka, R.; Martin, W.: Ceramic components and their implementation in industrial applications. CERAMIC APPLICATIONS **1** (2013) [1] 50–55

[2] Wötting, G.; et al.: Neue technologische Entwicklungen und Anwendungen von Komponenten aus Siliciumcarbid und Siliciumnitrid. cfi/Ber. DKG **84** (2007) D15–21

[3] Berroth, K.: Silicon carbide and silicon nitride ceramics for passive structural components in avionics, space and mechanical engineering. CERAMIC APPLICATIONS **2** (2014) [2] 42–46

[4] Firmenpräsentation FCT Systeme GmbH: Effiziente Sinteranlagen für die Produktion von Hochleistungswerkstoffen; cfi/Ber. DKG **89** (2012) [3] C100–102 (http://www.fct-systeme.de/de/content/Download_News_Presse/~nm.67~nc.69/Veroeffentlichungen-und-Presse.html)

[5] Kessel, H.U.: Sintered materials on the way to production by means of modern SPS technologies. cfi/Ber. DKG **86** (2009) [10] E145–154

[6] Berroth, K.; Martin, W.: Wear protection in powder processing with SSiC and Si_3N_4 components. cfi/Ber. DKG **81** (2004) [8] E16

[7] Frommherz, H.: Realisierung individueller innovativer Lösungen in der Misch- und Mahltechnik. cfi/Ber. DKG **85** (2008) D11–14

[8] Schnurpfeil, T., Reitz, M.: Tür auf für Siliziumkarbid. cav (2012) [5] 8