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"Silicon Nitride Ceramics for Product and process Innovations"

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Abstract.

During the last years large efforts were made, to get able to supply large and complex components made of gas pressure sintered silicon nitride. This opened new applications for such ceramic materials in ambient and very harsh environment and generated new markets for ceramic producers. The paper presents, a newly developed rapid prototyping routine for complex components as well as the properties of corresponding materials. Components for innovative avionic and space applications, dynamic materials testing, liquid metal processing, metal forming and mechanical engineering are shown. Not only unique properties of the material itself, but also newly developed and adopted shaping and machining technologies for this specific ceramics have let to highly valued products.
Due to its very specific set of material properties, silicon nitride has gained a lot of interest. New approaches in technical equipment were undertaken with corresponding research. However, except of seals, bearings and cutting tools, none of all expected high volume applications is industrialized. On the other side, a lot of less spectacular applications are state of the art today. They have opened a wide field for niche products and lead to technical solutions with less wear and corrosion but improved products.

Introduction

During the last years a large effort was made, to be able to supply even very large and complex shaped components made of sintered silicon carbide (SSiC) and of more likely of gas pressure sintered silicon nitride (GPSN) ceramics. This approach has opened new applications for such ceramic materials and also new markets for ceramic producers. On the other side, designers and engineers are now allowed to think much more complex in designing of ceramic components. In this paper, a new rapid prototyping routine for very complex components as well as the properties of the corresponding materials will be presented. Components for innovative optical aviation and space applications, for dynamic materials testing, for liquid metal processing, metal forming and chemical engineering are shown. Not only their unique properties, but also newly developed and adopted shaping and machining technologies for this specific ceramics have let to highly valued products.
By producing the housing structure of an infrared camera for aerospace with silicon nitride, higher resolution was reached due to a very low coefficient of thermal expansion, higher stiffness and less weight. Using silicon nitride, highly stiff, lightweight large scale structures for satellites can be realized with a material whit a very low CTE and improved outgasing behaviour. Also dynamic testing equipment mainly for avionic turbine blades can reach much higher frequencies and so reduce fatigue testing time and costs. With silicon nitride rollers for various functions, the lifetime of components in stainless steel and titanium rolling operations was extended by more than a magnitude, additionally improving the quality of the rolled wires, sheets, foils and thin walled structures. Improved efficiency of attrition mill without any contamination through to no metal

contact was reached with silicon carbide liners and silicon nitride agitators. Setters of highly strong, lightweight and corrosion resistance SSiC and HPSN led to triple the capacity of sintering furnaces for high volume technical ceramic parts and also reducing the specific energy consumption. As a result it can be stated that the economic availability of new advanced non oxide ceramic materials can generate new technical solutions for avionics, space and industrial equipment and processes.
Due to their very specific set of material properties, silicon nitride and silicon carbide have gained a lot of interest in the past 20 years. Moreover, many new approaches in technical equipment like gas turbine and motor engines, cooking systems, seals and bearings, cutting tools, metal pumps, electronic substrates and many others were put in place with corresponding research activities.
On the other side, a lot of less spectacular applications are state of the art in industry today. They have opened a wide field for niche products and lead to new technical solutions with higher performance, extended service time, less wear and corrosion and improved product properties.

1 Material and Fabrication

FCT Ingenieurkeramik has established material grades for GPSN and SSiC as well as for some composite Materials like C_f/CSiC and the corresponding processing routes for the commercial fabrication of a broad range of components with mainly large and complex sizes and shapes and i. g. very narrow tolerances.
Standard grades for GPSN are composition of 90% down to 97% of Si_3N_4, with corresponding sintering additives which are sintered at 1 MPa of nitrogen gas pressure or using our HP or SinterHIP process at up to 1850°C and 200 MPa for improved properties.

As major fabrication routines we use:

1. slip casting for complex, thin walled components
2. cold isostatic or uniaxial pressing of preforms
   with
3. subsequent green machining,
4. sintering, gas pressure sintering, HP or SinterHIPing
   and if necessary but most likely for narrow tolerances
5. final machining by laser cutting, grinding, honing, drilling, lapping and polishing.

Rather large components with one dimension up to 1,25 m in length and/or 0.5 m in diameter, but also tiny things with only some mms feed into our product range. We typically start to produce prototypes according to customers design requirements but also do small and intermediate series up to 10,000 pieces per year or fabrication lot, whenever prototypes were successfully tested.

2 Applications

2.1 Light Metal Cast House Applications

One main application area has been located and developed in light metal casting.
Here mainly two of the materials properties are required:

1. excellent corrosion resistance against metal melt
2. excellent thermal shock resistance
   additionally helpful is
3. high strength and fracture toughness

Components like thermocouple sheaths, heater sheaths, riser tubes, valve seats and plungers, degassing agitators and a lot of other very specific parts are in use or are tested in new advanced casting systems. The size is medium to large, the complexity is simple to medium. Rather rigid components are required for the rough working conditions in foundries

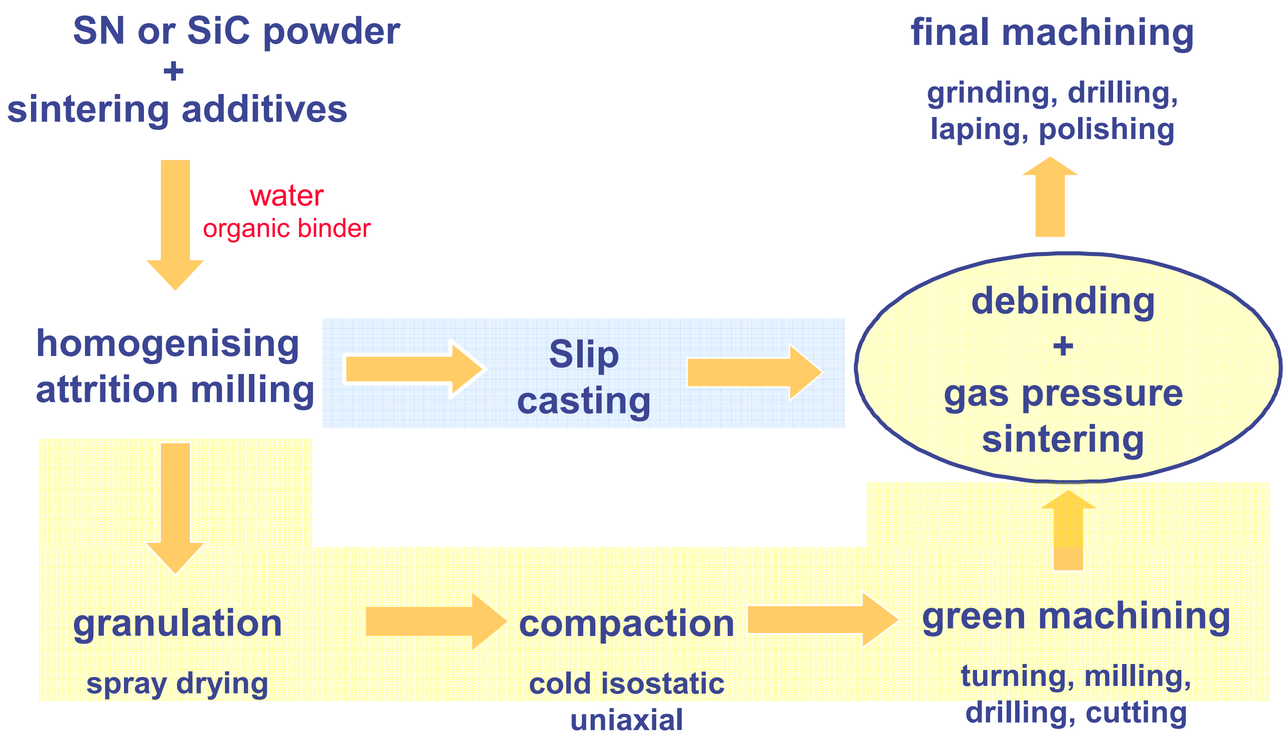

Fig. 1 production sketch

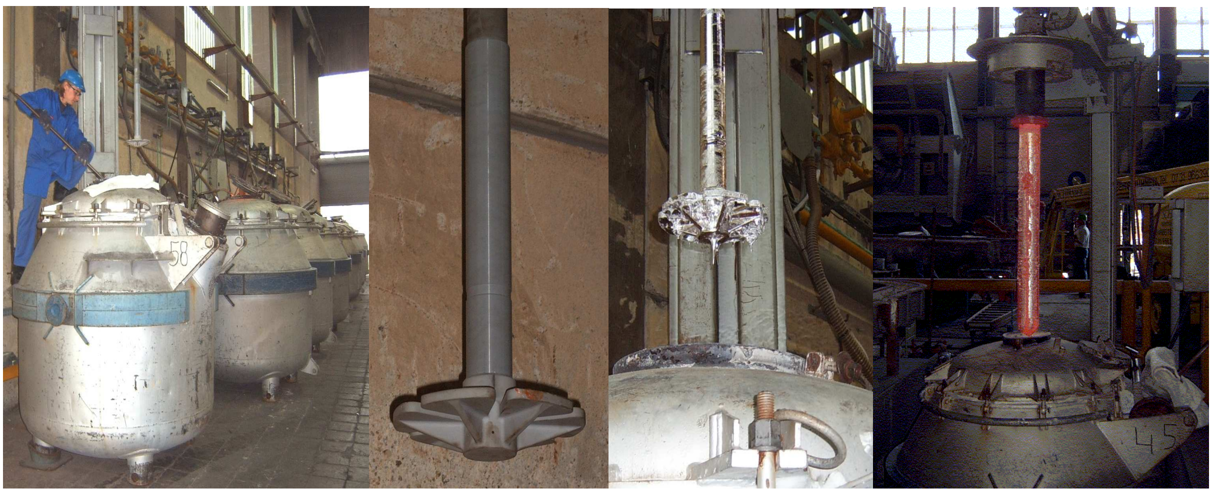

Fig. 2_Example for foundries: degassing impeller and immersion heater sheath for a transport Aluminium tank

2.2 Metal working

2.2.1 Rolling

In metal industry, new shaping approaches for even complex part are rolling operations. For such shaping processes steel, WC and ceramic rollers are used. Silicon nitride due to its excellent thermal

shock behaviour, high strength, hardness and toughness shows improved service time and product quality in both, at cold and even at hot rolling with temperatures up to 1050°C in rolling of stainless steel, titanium and aluminium alloys.
Further advantages are the possibility of cooling reduction and the lack of material transfer by friction welding to the rolled metal.
Even more successful are wear parts in metal forming facilities, like guiding, drive and break rollers where we could extend the service time by more than a magnitude.

Fig. 3 spin rollers for wheels and strip rollers with silicon nitride ring and barrel for Al-foil

2.2.2 Welding

For the production of welded tubes and profiles, highly wear resistant and tough calibration rollers have become state of the art because of their very much extended service time and accuracy.
Also for the fixation and precise, reproducible alignment of steel sheets, fixation pins are widely used in automotive industry. The advantage of silicon nitride in welding is the improved strength, thermal shock capability and the non wetting for sparks created through the welding process.

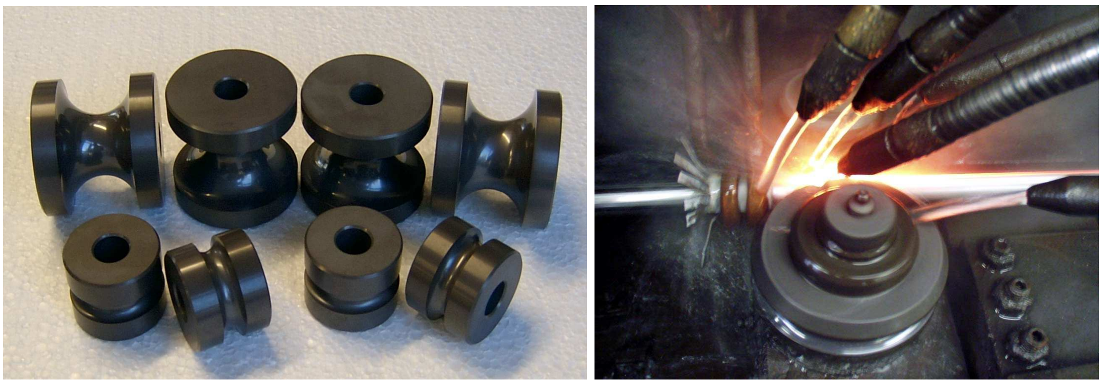

Fig. 4 guiding and welding rolls and welding caliber rolls in service

2.3 Mechanical and Chemical Engineering

In mechanical engineering, many applications need a wear resistant material which also can withstand a certain impact stress or mechanical load. The material preferably has to have a high thermal conductivity, corrosion and erosion resistance. This is perfectly met by SSiC with a thermal conductivity of 140 W/mK and universal corrosion resistance, but also GPSN with a thermal conductivity comparable.to stainless steel and ranging up to 70 W/mK for special grades as well as very good corrosion resistance and improved strength and fracture toughness makes it candidate for many applications in this field.
Highly precise sleeves for calendering equipment with slits down into the µm range are state of the art for high efficiency dispersion in colour industry. Agitator arms and rotors as well as a broad

variety of shapes and dimensions of lining inserts in mixers and attrition mills are other typical examples for this industry. Liners can be made monolithic with about 500 mm in diameter and up to 1 m in length with GPSN and SSiC.

Fig. 5 GPSN roller sleeves for calendaring, GPSN mill barrel inserts and GPSN agitators for attrition mills

2.4 Optical Engineering

For optical applications, mainly for instruments which are used in airplanes or space, the density is very important beside stiffness, coefficient of thermal expansion and long term durability. Also the mechanical strength and fracture toughness is important, because on starting and landing, very high mechanical loads can appear. Another point is the thermal conductivity, which sometimes should be as high as possible and sometimes as low as possible.
Here with a sophisticated green machining and sintering technology, very large and complex structures for optical equipment like space telescopes and avionic cameras have opened a new promising application. Also long term stable guiding beams for wafer steppers and optical measuring systems as well as supporting structures for lenses, can be either made from silicon nitride or silicon carbide, depending on the best fitting property set.
One of the most sensitive parameters for large optical components is the CTE. At room temperature, for silicon nitride a value of 1x 10^{-6} /K is the lowest available value in combination with low density, high stiffness, strength and durability. The CTE of corresponding materials for optical use is shown for the temperature range between -100°C and +100°C.

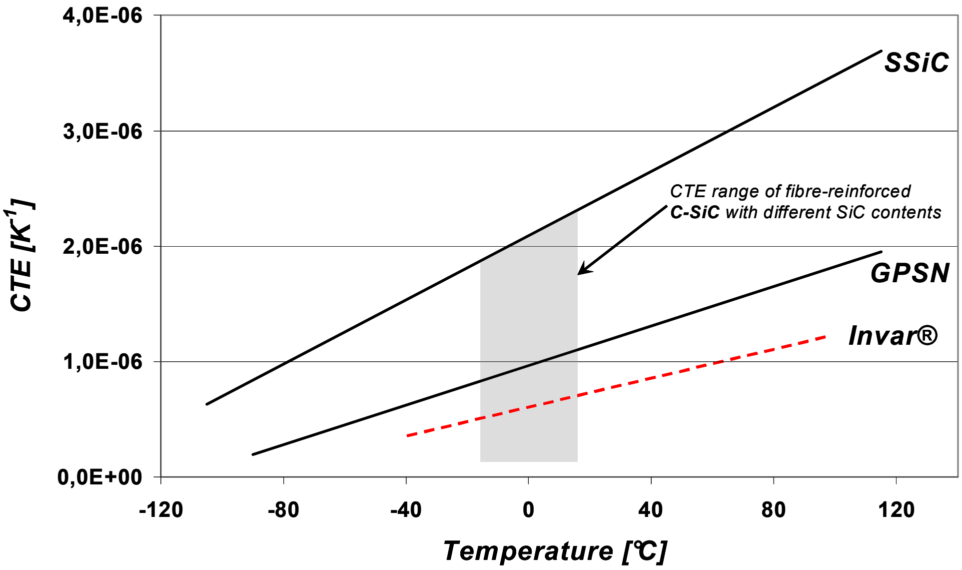

Table 1 Coefficient of thermal expansion CTE for structural “optical” materials

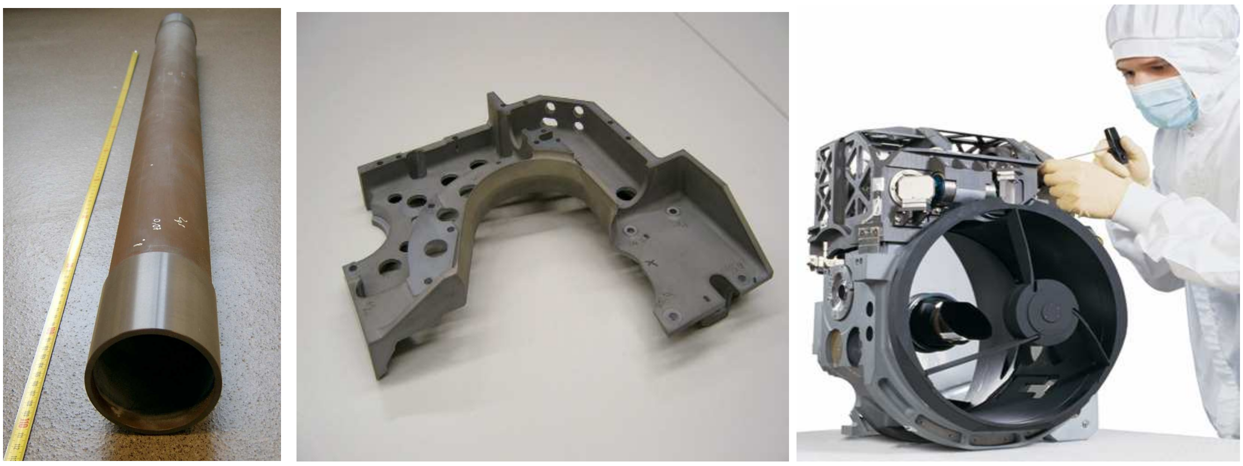

Fig. 6 lightweight satellite beam, instrument carrier and ceramic structure for avionic camera at final set up in clean room environment

2.5 Electrical Engineering and Electronics

Also in electrical engineering, silicon nitride components have gained specific application niches. Highly strong, thermo mechanical and thermal shock resistant insulator components show improved service behaviour compared to alumina or porcelain. Coil supports for inductive heating equipment for metal heat treatment have tripled the service time. Heater separation and shielding plates as well as drive rollers which withstand temperature and corrosive atmosphere e.g. for thin film solar cell production have improved the performance of CVD and PVD coating equipment. Power feed troughs for vacuum and high pressure furnaces can better withstand the thermal gradient which occurs in this application.
In electronics, high stiffness and low wear is reached for guiding beams and supporting plates for wafer machining and handling. Also probe cards in chip manufacturing and testing equipment has gained some importance and reasonable market volume.

Fig. 7 supporting plate for wafer handling in chip production plants, thickness 1 mm, diameter up to 400 mm

Another approach can be made with composites, based on silicon nitride having a rather high electrical conductivity. Wear resistant EDM tools and Sensors can be fabricated as well as non metallic heating elements for temperatures up to 1400°C. One advantage of such heater is in fact, the possibility to keep them very flat and up to about 400 °C, they do not deform or build up stresses even if only heated from one side, due to their low CTE.

2.6 Medical Application

screws and bone plates are tested as replacement for alumina and zirconia in order to provide higher strength and lower density with lightweight elements for lower Xray shadowing in post operation inspection and additionally improved bio combatibility for implants.

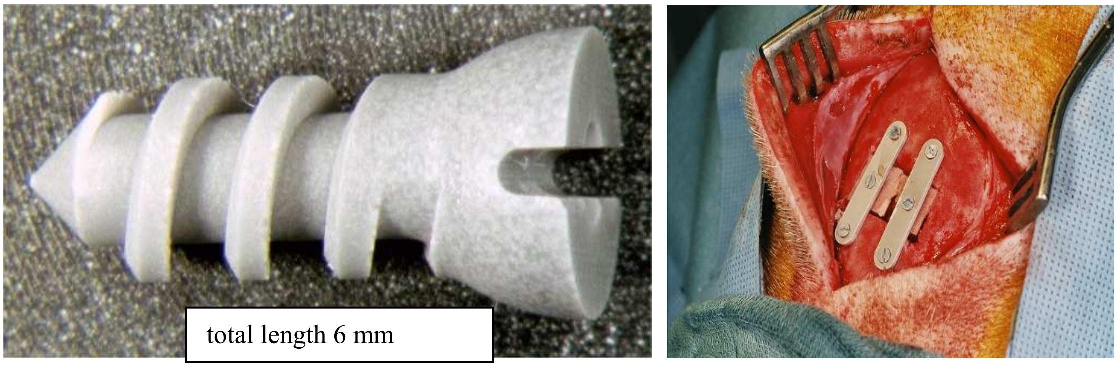

Fig. 8 surgical screw for implants and dummy implant with GPSN fixation screws implanted into a pigs head

2.7 Thermal Engineering

In high temperature and thermal engineering, the demand for highly strong and long term corrosion resistant kiln furniture for technical ceramics as well as for sanitary and tableware has grown in the last years. This was strongly pushed by the use and implementation of robotic set up and unloading of high volume ceramic parts. Here a high purity HPSN or HIPSN as a dense and high strength material with good thermal shock behaviour and excellent corrosion resistance has gained niche markets. Using HPSN setters as shown in picture 9 in a specific application, the capacity of the furnace was tripled and investment in additional equipment could be avoided.

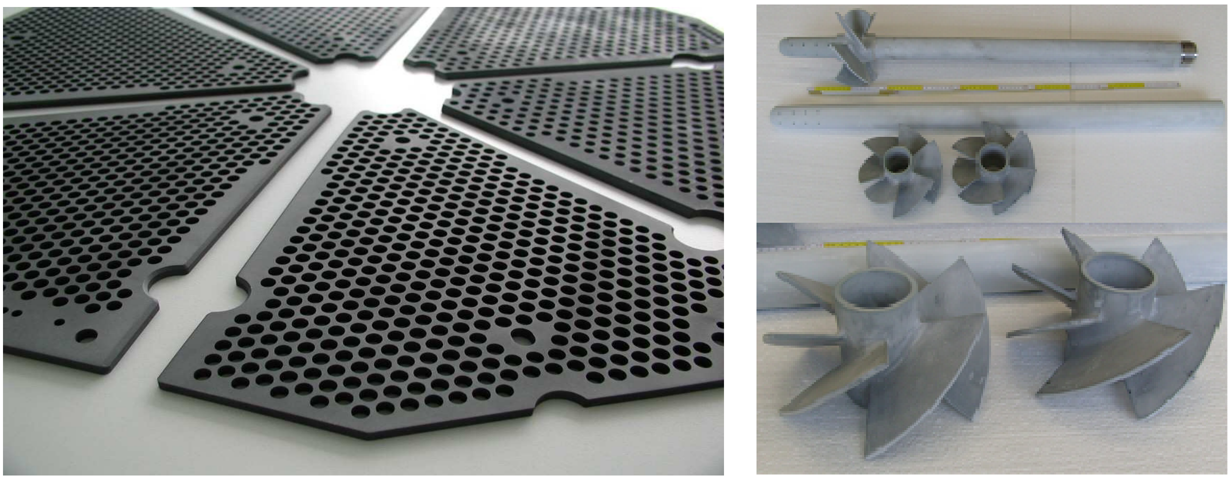

Fig. 9 HPSN setter plates for high volume firing technical ceramics, RBSN high power gas burner components

2.8 Testing Equipment

Due to it`s low density, high Youngs modulus, long term mechanical stability and the high strength and fracture toughness as well as it`s electrical insulating behaviour, silicon nitride is a candidate material to be used as a swinger head in high frequency vibration test equipment. A further

advantage is it`s very low CTE, leading to no dimensional change within a certain temperature range and so keeping a stable test frequency. This enables silicon nitride to be used as calibration vibrator for frequencies up to 25 kHz.
The complex shaped and finely structure component with very narrow tolerances and minimal wall thickness of 0.25 mm was developed in tight cooperation with TIRA, a world leading company for special testing equipment.

Fig._10 swinger head, external and internal structure and calibration shaker unit for 25 kHz

3 Improved Properties

As mentioned above, silicon nitride shows improved application benefits compared to other ceramics or metallic solutions. Mainly in wear and foundry industry, solid silicon nitride components gives a much longer service time compared to solutions with coatings. Even more improved properties can be reached by silicon nitride based composite materials.
Here, a wide variety of different additives help, to tailor materials for specific use in new advanced technologies.
With the addition of TiN for example, the hardness and the electrical conductivity can be adjusted to a wide range of technical requirements. Oxide and non oxide sintering additives can help to change and improve the corrosion resistance by the reduction of wetability for metal melts. Also the thermal conductivity can be influenced by special rare earth oxides. A wide range of possible compositions is still under investigation.

4 Conclusion

In the paper it is shown, that a wide range of applications of silicon nitride ceramics have been achieved due to the availability of components with different sizes, shapes and complexity. Small and very precise as well as rather large and complex components can be reproducibly and economically produced with a high standard of quality. Materials and components are highly reliable, also in very though applications. New technical solutions are though possible for highly advanced processing equipment and routes.

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