Selective Laser Sintering as an Additive Manufacturing Method for Manufacturing Ceramic Components

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Abstract

Selective Laser Sintering (SLS) offers the advantage of directly manufacturing complex-shaped parts without the uneconomic loss of material so common when conventional manufacturing methods are applied. Originating from the laser sintering of polymers, lots of research has been conducted on advanced materials like metals and ceramics. In recent years the Fraunhofer IKTS has conducted research on the laser sintering of SiC and different hydroxyapatite (HAp) powders. The HAp powders have been characterized based on their laser sintering ability with the variation of process parameters like spreading behavior, different grain shape and size, hatch clearance, etc. More elaborate research has been conducted concerning the laser sintering of SiSiC components. After infiltration and pyrolysis of laser-sintered SiC parts, the resulting SiSiC components showed similar mechanical characteristics to those of conventionally processed SiSiC components. Different complex geometries were realized e.g. a 120-mm honeycomb-structured mirror support model (wall thickness of 1 mm, for aerospace applications) and a small turbine rotor (minimal wall thickness of 0.3 mm) as a demonstrator part for automotive components. A laser mirror with integrated cooling channels for application in movable beam arms for laser material processing was also manufactured. Another successfully generated SiSiC component is an injection molding tool insert (ca. 35 x 30 mm; diameter x height) with a complex-structured surface and inner cooling channels. The next steps will be towards research dealing with more complex designs and other ceramics as well as metal-ceramic hybrids.

Keywords: Selective Laser Sintering (SLS), ceramic, silicon carbide, hydroxyapatite

I. Introduction

First conceived by Householder [1] and further evolved by Beaman & Deckard [2], Selective Laser Sintering (SLS) is used as a possibility to directly process powders layer-wise to form a single product. According to the contours of a 3D Computer-Aided Design (CAD) model, a laser source e.g. CO_2 (alternative: lamp/diode pumped Nd:YAG, disk or fiber lasers [3]) of a SLS device fuses the particles of a specific powder together. Then a new layer of powder is deposited over the generated contour and irradiated by the laser again, according to the given dimensions of the CAD model. In this way, layer by layer, complex-shaped components are built up. This now well-known concept is one of the most advanced processes for manufacturing complex metallic and polymer parts. However, it is becoming more and more important for ceramic materials [4–11] or composites like cermets [12].

All of these material and process alternatives are driven by the huge potential of Additive Manufacturing (AM) for the direct manufacture of complex-shaped parts of all types, which either cannot be produced at all with conventional manufacturing methods like milling or grinding or only with a severe loss of resources and energy. With regard to advanced materials like ceramics, depending on the starting material and specific structure, possible applications range from biocompatible and bioactive parts consisting, for example, of calcium phosphates like hydroxyapatite (HAp) [13–17] and/or polyether ether ketone (PEEK) [18–19] to wear- and friction-resistant parts made of SiC [20]. As seen in the following sections, the flexibility of production and design of SiC components given through AM offers huge application potential.

However, this additive-produced material needs to be critically compared with the material properties of conventionally processed ceramics. The presented paper shows the application spectrum of selective laser sintering for the fabrication of biomaterials as well as complex-shaped wear-resistant SiSiC components. In doing so, it demonstrates how the material properties are improved by means of targeted material selection and process optimization.

II. Equipment and Reviewed Materials

One important aspect regarding the SLS process of ceramic powders is the problem that pure ceramic powders do not simply consolidate in a solid-state sintering process. Instead, a liquid phase is required to densify and consolidate a laser-sintered ceramic structure. Low-melt-

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ing organic additives [21] or a eutectic composition of two ceramic phases melted together with a laser beam to a uniform ceramic matrix structure [22] are possible requirements. In addition, inorganic *in situ*-produced SiO_2 can function as binding phase as well as glass [23]. However, actual sintering of the ceramic particles usually does not occur or only under very intense conditions [22].

Therefore the SLS is often directed at the production of porous ceramic structures. In the following section, this brief overview of the research at the Fraunhofer KTS will give an insight into the research conducted with two totally different materials boasting a wide field of application when manufactured with selective laser sintering.

All laser sintering experiments in this paper were executed with an EOSINT M 160 (Co. EOS GmbH, Germany) featuring a water-cooled infrared CO_2 laser with the wavelength $\lambda = 10.6$ µm. The laser's velocity spans from v = 50 – 10 000 mm/s the hatch clearance being adjustable between h = 0.01 – 40 mm. With a maximum power of up to 100 W, polymers, metals and ceramics are potential processing materials.

(1) Laser sintering of HAp

Against the background of the current demographic change, artificial bone implants and biocompatible materials are becoming more and more important. Biomaterials that enable tissue material to grow into a component have one thing in common: a porous structure. Since the SLS of ceramics produces porous structures *per se,* the manufacture of such biocompatible structures is the obvious step. A suitable ceramic material is hydroxyapatite. However, porous HAp structures do not have sufficiently high compressive strength to be used as long-term implants. But in combination with the likewise biocompatible and most of all significantly stiffer and more rigid PEEK, an application as a long-term implant might be possible.

Now, in a first experimental setup concerning HAp, the objective was to laser-sinter pure granules without the need for organic binders. In a second experimental setup, a certain amount of PEEK is added to act as a strengthening component and gluing agent between the HAp particles.

The grain size in addition to the shape of the desired starting material is known to have a significant impact on the spreading behavior of powders during layer-wise deposition. Spreading tests were conducted and resulted in the selection of suitable HAp powders with specific and widely differing grain size (determined with a Mastersizer 2000, Co. Malvern Instruments, UK) as seen in Table 1.

Lots of different parameters for the laser sintering were tested but, as anticipated, the laser sintering of pure HAp granules proved almost impossible. The coarser HAp 1 did not melt at all. Only the finer HAp 2 and 3 powders (Fig. 1) partially consolidated if only into melting beads. The laser parameters such as laser power (P) and velocity (v) as well as the hatch clearance (h, distance between the beams focus point) were varied accordingly (Table 2). In addition, the SLS process was accompanied by "vaulting" HAp granules. This is an effect of the direct laser energy input on the pure particles, which are unable to absorb the energy fully and are pushed aside instead.

Therefore, in the second experimental setup, mixtures of hydroxyapatite (type HAp 3) powder and PEEK (150 XF/PF) were prepared and evaluated with regard to the bonding or gluing effect of the polyether ether ketone. The amount of PEEK was varied between 1 and 40 wt%. Then, for the most promising combinations the laser sintering parameters were adjusted. In the end, the best continuous single layers were achieved with a PEEK amount of 40 wt% in the hydroxyapatite powder type HAp 3. (Figs. 2 and 3). As seen in Fig. 2, the scanning electron microscopy (DSM 982, Leo, Germany) shows the hydroxyapatite particles bonded firmly together in a molten network of polyether ether ketone.
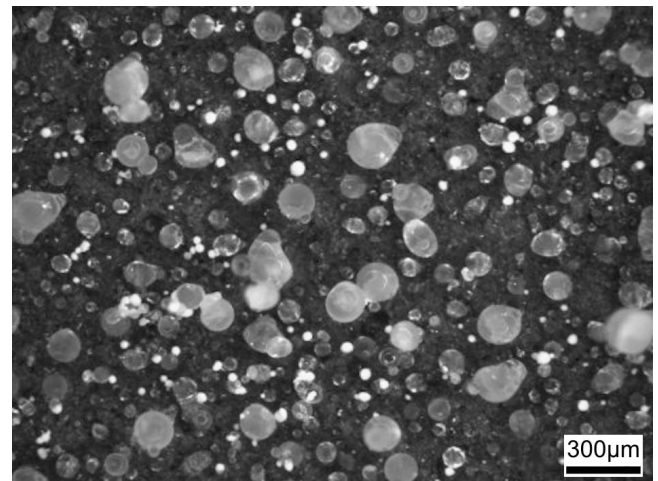

Fig. 1: Mixture 3 (HAp 2) molten beads.

Table 1: HAp/PEEK powder grain sizes.

Particle size	HAp 1 (50752) [1]	HAp 2 (50754)[1]	HAp 3 (50408)[1]	PEEK 1 (150XF)[2]	PEEK 2 (150PF)[2]
d_{10} [µm]	44.6	2.0	10.8	–	–
d_{50} [µm]	71.4	31.0	32.8	25.0	50.0
d_{90} [µm]	114.0	76.2	61.5	–	–

1) Ceram Ingenieurkeramik GmbH, Germany
2) Victrex, Europa GmbH, Great Britain
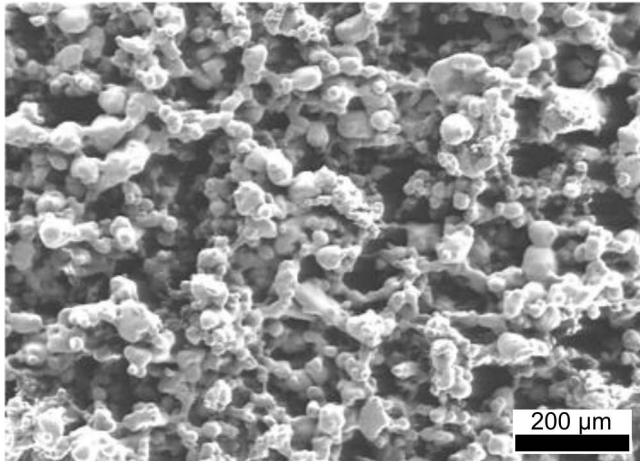
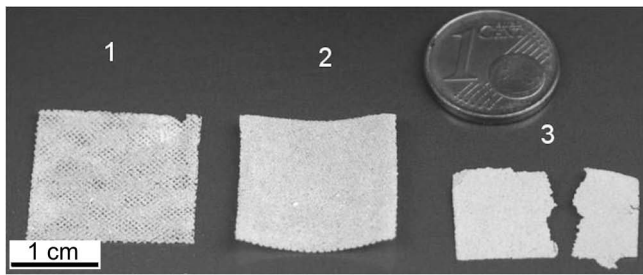**Fig. 2**: Microstructure of a laser-sintered HAp-PEEK layer

Fig. 3: Continuously laser-sintered HAp-PEEK layers.

Table 2: Process parameters of laser-sintered pure HAp powders.

Experiment	P [W]	v [mm/s]	h [mm]
M1 (HAp 2)	4	50	0.08
M3 (HAp 2)	8	50	0.08

The working laser parameters are shown in Table 3.

Table 3: HAp-PEEK mixtures and corresponding laser sintering parameter to form single layers.

Sample	P [W]	v [mm/s]	h [mm]
1	3	4000	0.03
2	3	3000	0.03
3	3	3000	0.01

Sample 1 does have an even surface but is not as densely consolidated as sample 2 with the same hatch clearance but lower laser velocity/scanning speed. Decreasing the hatch clearance demands careful handling afterwards as the layers became more brittle.

(a) Conclusion and outlook

The first experimental setup (different hydroxyapatite granules) has shown that it is at least possible to transfer pure HAp granules to a temporary liquid phase, resulting in a loose network of HAp beads. However, to achieve a continuous layer of this biocompatible material, an organic binder was needed. Concerning the second experimental setup, using the likewise biocompatible PEEK, single layers of HAp were finally achieved. An amount of 40 wt% PEEK 150 XF and 60 wt% hydroxyapatite powder 50408 (type 3) proved the best composition. However, owing to a lack of a sufficient number of layer samples, only qualitative statements regarding their mechanical strength were possible. The samples do have a certain tensile strength but are too brittle. Owing to the low layer thickness, measurements of the compressive strength were not possible, but a punctual force can be applied without the layer being torn apart.

In a next step, laser-sintered HAp-PEEK samples have to be evaluated regarding their biocompatibility and bioactivity. But to achieve that goal, the laser sintering process has to be improved. Hydroxyapatite-PEEK mixtures with better spreading behavior might prove useful, but the way the two biocompatible materials are mixed together also has to be improved. Instead of the HAp particles just being mixed, HAp particles coated with PEEK might prove better. The advantage of that method would be a smaller amount of polymer and a larger amount of the bioactive component. The sintering ability should be better when compared to a physical powder mixture. In that way, by having just one, preferably spherically shaped material, the apparent density and in the end the density of the laser-sintered part could be improved. Better handling ensures the possible use as a complex-shaped implant.

(2) Laser sintering of SiC

As mentioned in the introduction, one of the main questions is whether or not laser-sintered parts can made comparable with conventionally produced parts with regard to their mechanical strength. The huge advantage of selective laser sintering is the production of complex 3-dimensional parts from the beginning. But achieving dense and tough ceramics is a totally different matter.

Now, to achieve such 3-dimensional laser-sintered structures, ceramics can be consolidated with miscellaneous post-treatment processes, for instance reaction infiltration or glass infiltration. A typical example is the material SiSiC (silicon-infiltrated silicon carbide)[24–25]. Conventionally produced SiSiC exhibits high thermal resistance, hardness and strength and thus offers enormous application potential. For achieving laser-sintered SiSiC components, the following steps are well-known: first the shaping of a porous SiC body followed by pyrolysis. As result, a porous SiC/C composite is obtained owing to the partial decomposition of SiC to Si, forming SiO_2 and carbon residues originating from organic additives. In the second step the composite is subsequently reaction-infiltrated with silicon. The result is dense SiSiC ceramic. In this case the free carbon produced during the pyrolysis of organic additives is used as a binder in the ceramic shaping process. Alternatively, the processed porous SiC body can also be infiltrated additionally with a carbon source prior to the pyrolysis. Thus, during reaction infiltration and above the melting point of silicon, the free additional carbon reacts with the silicon to form even more secondary SiC. The pure silicon fills the pores. The resulting SiSiC

ceramics exhibit no shrinkage during thermal processing, ensuring high dimensional accuracy.

(a) *Procedure and challenges met*

A laser-sintered structure has to be porous and also tough enough to be handled properly. For the following research, two different SiC powders were chosen because of their good spreading behavior when deposited as single layers. The powders F 360 and F 500 (ESK SiC GmbH, Germany) differ in their grain size and packing density. Particle size distribution measurements determined the d_{50} of F 360 at 23 µm and for F500 at 13 µm (determined with a Mastersizer 2000, Co. Malvern Instruments, UK). The bulk density was determined at 1.40 g/cm³ for the silicon carbide F 360 and 1.27 g/cm³ for the F 500. In order to evaluate varying laser parameters and their effects on the materials density and compressive strength, bending bars (7 x 7 x 70 mm³) were made and evaluated to optimize the parameters. However, a lot of problems can be encountered during the production of such parts. No matter how easy the geometry, one of the most important aspects for selective laser sintering is the control of defects like delamination or the formation of cracks caused by thermal stresses in the part during processing (Fig. 4).
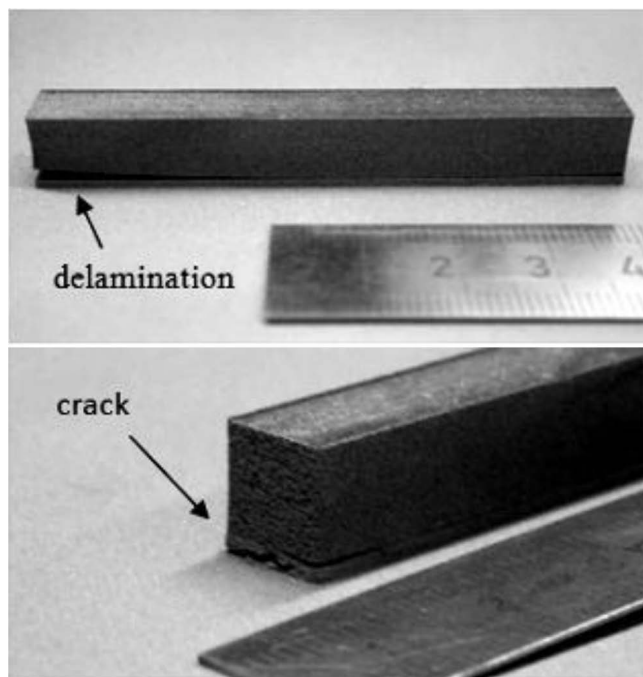

Fig. 4: Defects in test-bar samples after the laser sintering process.

The reason for delamination or the formation of cracks is the temperature gradient between the residual heat of the part and the heat caused by the laser impact. If the area of the residual heat lies above the neutral bending axis, higher stresses will occur as a result of heating and cooling cycles. If these stresses are higher than the strength of the part itself (connection between support/base-plate and support/part), delamination and cracks are the consequences (Fig. 5). The upper layer areas which lie in the influenced heat zone expand whilst heating and shrink whilst cooling. The lower layer areas do not lie within the influenced heat zone. In doing so, they act as resistance against thermal expansion. This effect is referred to as disabled shrinkage.

Consequences are delamination and formation of cracks. With suitable heating in the process or a reduction of heat input (to both, in part and support), these defects can be prevented.
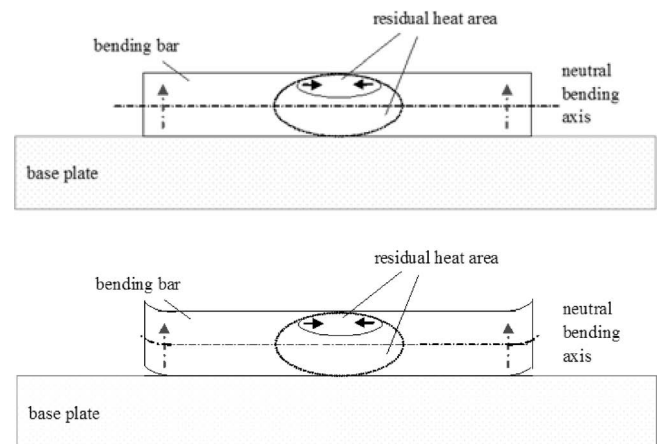

Fig. 5: Reasons for defects (delamination and cracks) occurring during selective laser sintering.

As mentioned before, the infiltration of liquid Si yields secondary SiC as a result of the reaction of carbon with Si. It is also possible to add a certain amount of additional carbon by infiltrating the porous SiC structure with resin. In effect that means the more carbon in the part the more secondary SiC in the dense structure. In that way the carbon-enriched structure will become denser and should provide more mechanical strength.

So, after the porous bending bars had been laser-sintered, they were infiltrated for 10 – 15 minutes at a reduced pressure of 35 mbar with the resin Phenodur VPW 1744/60 WA (Co. SOLUTIA), which was then pyrolyzed at a temperature of 1200 °C in an argon/N_2 atmosphere. Now that carbon-enriched part is ready to be infiltrated with silicon. The laser-sintered part is placed onto a crucible in an infiltration oven. On the inside of the crucible is a porous SiC or carbon support. That sintering ring is then filled with a certain amount of pure silicon. Now in a first step the oven is heated up to 1000 °C under inert conditions (argon). From 1000 °C to 1400 °C (the silicon is now molten) the oven is further heated under vacuum. In that temperature range, the surface of the laser-sintered SiC part is activated. As it has now lost its outer SiO_x skin (as result of the processing conditions), the SiC component is now wettable with liquid silicon. Because of capillary forces, the molten silicon infiltrates into the pores of the part. Now the secondary SiC is formed (Fig. 6). Once 1600 °C has been reached, the cooling phase starts. The result is a fully infiltrated and dense SiSiC part.

Finally, with just the right amount of carbon (10 wt%) and the finer starting material (SiC F 500, $d_{50} = 13$ µm), it was possible to produce laser-sintered SiSiC parts that are very similar in their mechanical behavior to conventionally produced SiSiC parts (Table 3). The ideal laser parameters for that optimized silicon are in an interval of the laser power between 45 – 53 W, scanning speed of 200 – 300 mm/s and a layer thickness of 50 µm.
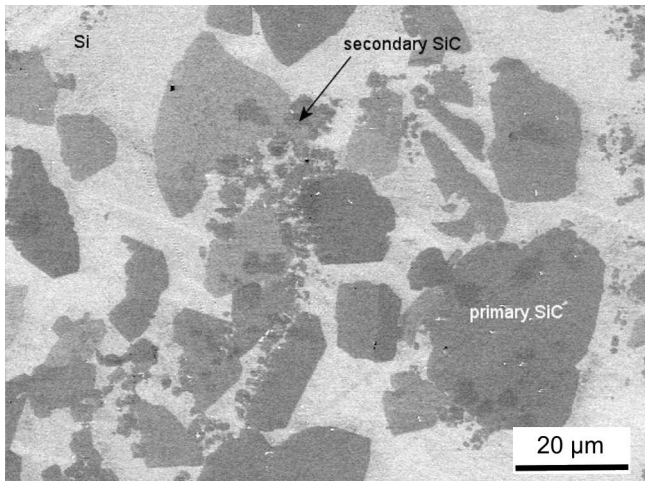
Fig. 6: The different types of the existing silicon carbon after the final Si-infiltration.

Furthermore, the excellent handling ability of the laser-sintered parts allowed intense surface treatment even down to a few nanometers.

Table 4: Mechanical properties of SiSiC batches, laser sintered (A - C) and conventionally produced SiSiC (D).

	A	B	C	D
Density [g/cm³]	2.65	2.79	2.85	3.05
Flexural strength, (4-point, room temperature) [MPa]	196	223	267	300
Young's modulus [GPa]	205	245	259	320

A: SiC with 23 μm particle size, without carbon doping
B: SiC with 23 μm particle size, 10 wt% carbon doping
C: SiC with 13 μm particle size, 10 wt% carbon doping

On the basis of these experimental results and finally with material properties comparable to conventionally produced parts, lots of different geometries were realized. These illustrate the versatility and potential of the laser sintering with regard to their potential use in different applications.

III. Results

Especially parts with curvatures are a challenge that laser sintering is able to master. Accordingly, with the optimized parameters, a small turbine rotor was laser-sintered as possible demonstrator for automotive applications. The thickness of the sidewall could be reduced down to 300 μm (Fig. 7). Owing to its delicate structures, the turbine rotor needed a metal plate as support structure.

Another successfully built ceramic part was a laser mirror with integrated cooling channels in the dimensions 55 x 40 x 12 mm³, with a surface roughness of $R_a = 20 - 30$ nm after lapping (Figs. 8 and 9). This part was made for application in moveable beam arms for laser material processing [26]. The reduction in the mass of the mirror would lead to higher acceleration. In essence, thanks to the savings in terms of process time and design, the mass of the mirror was reduced to an eighth of that of a conventionally used copper mirror.

Fig. 7: Laser-sintered SiSiC turbine rotor.

Fig. 8: SiSiC mirror with inner cooling channels during laser sintering.
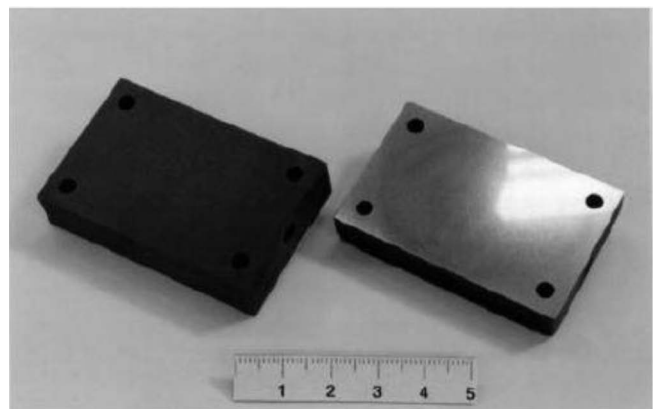

Fig. 9: Finished laser-sintered SiSiC mirror with inner cooling channels.

Thanks to the excellent thermal behavior of SiSiC (high thermal conductivity, low coefficient of thermal expansion), this material can be used in the harsh conditions of space. To build a support for aerospace mirrors, a specific carrier was manufactured (Figs. 10 and 11). This 120-mm-diameter part consists of a honeycomb structure with a wall thickness of about 1 mm [27].
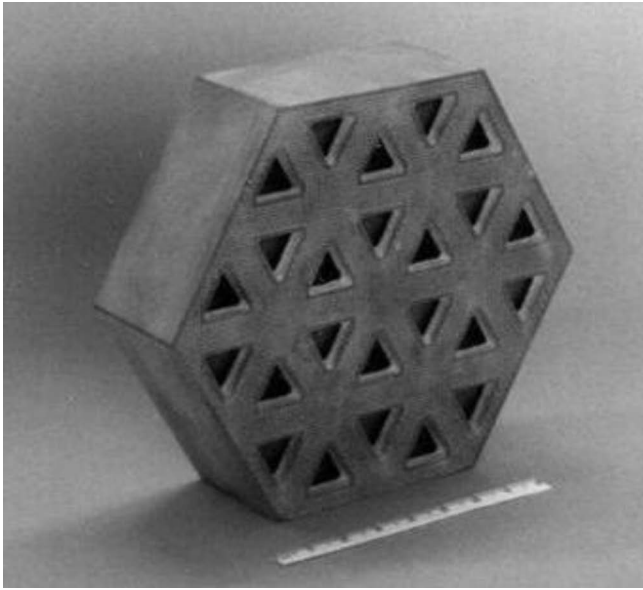
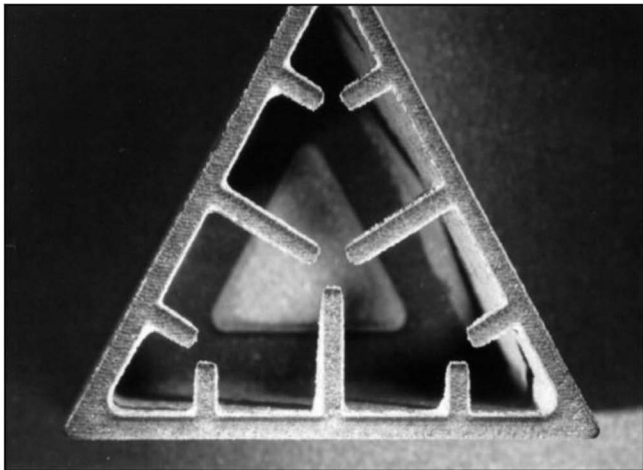Fig. 10: SiSiC model of a laser-sintered mirror support.

Fig. 11: Honeycomb structure of the mirror carrier (1-mm wall thickness).
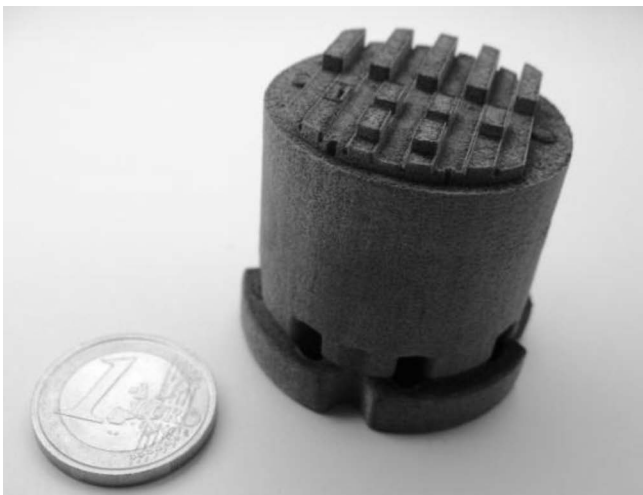

Fig. 12: SiSiC tool insert for injection molding.

As mentioned earlier, owing to its wear-resistant characteristics, SiSiC is also an excellent material for use in wear- and friction-intensive applications. In this context, an injection molding tool insert (Fig. 12) with in-

ner cooling channels (Fig. 13) was laser-sintered (dimensions: 35 x 30 mm; diameter x height). Now the lifetime of such an insert can be enhanced significantly.
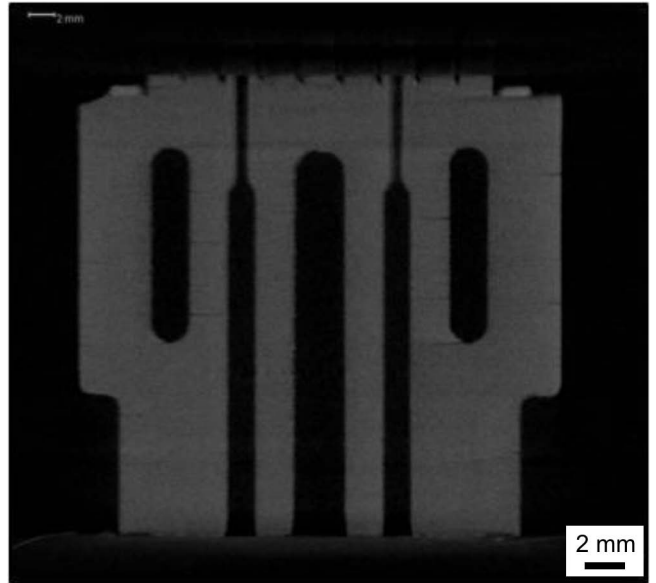

Fig. 13: CT cross section image of the inner cooling channels of the PIM insert.

In a next step and with the combined efforts of many participants [1], promising results were obtained with the combination of conventional laser sintering and laser micro-sintering [28–29]. Background for this development is the known proportional increase in time with the decrease of the layer thickness. But exactly that and the use of fine powders are requirements for an excellent surface quality, filgree details and, with regard to ceramics, also high mechanical toughness. To economically manufacture large parts nevertheless, a hybrid process was developed combining a large experimental setup and coarser starting material (conventional SLS) for large volumes with a small-scale setup and ultrafine powders in order to achieve very delicate parts (laser micro-sintering).

As result, large complex-shaped structures were manufactured by means of "macro" laser sintering with layers of at least 50 μm and additionally provided with filigree details formed by the laser micro-sintering with lower layer deposition of 10 μm (Fig. 14). Thus, with the use of finer SiC powder for the micro-part, the resulting micro-component was "connected" to the former mentioned tool insert. Both parts were then joined during the high-temperature reaction infiltration process, resulting in one laser-sintered SiSiC hybrid tool insert (Fig. 15). Now, "this could be a future concept for cost-effective production of laser-sintered SiC components" [25].
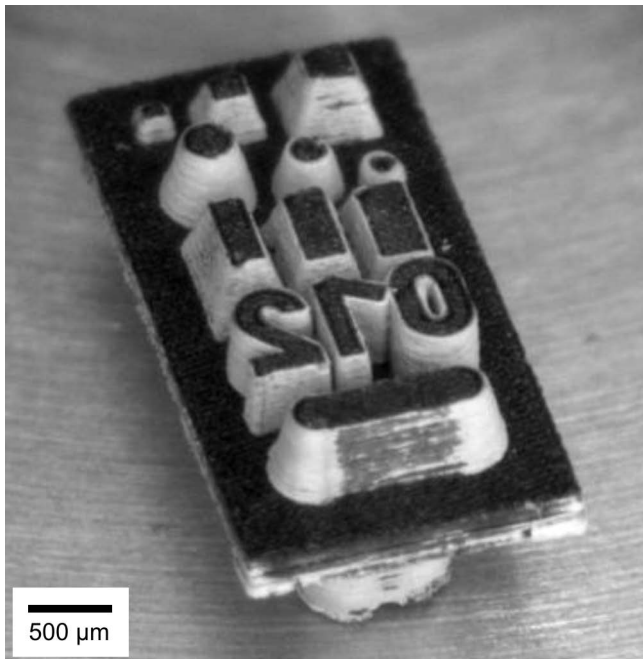
Fig. 14: Micro-laser-sintered part (University of Applied Science Mittweida).
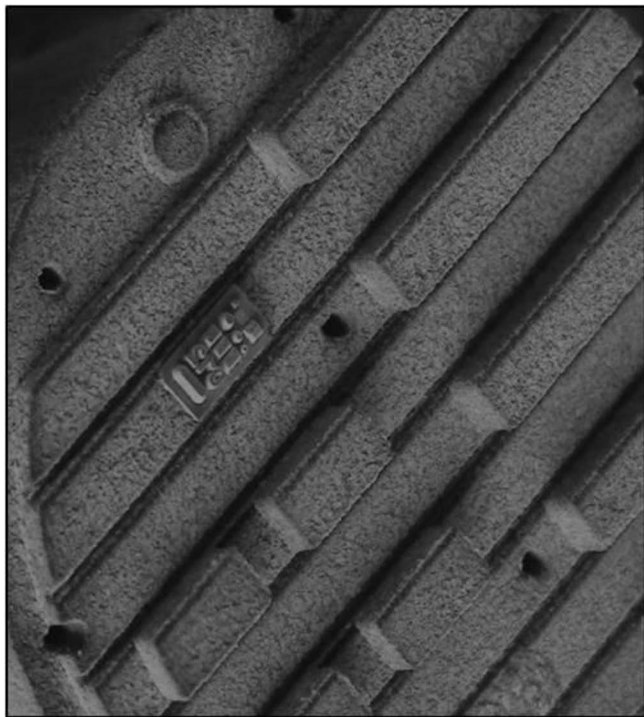

Fig. 15: Laser-sintered hybrid tool insert made of SiSiC.

IV. Conclusion

Selective laser sintering technology not only has huge potential, it is a sophisticated processing method for specific and well-understood materials and elaborated process optimization. The laser sintering of SiC followed by post-processing to dense SiSiC components can produce complex-shaped functional components for a wide variety of applications requiring wear-, friction- and thermal resistance. As shown in the above sections, material and process optimization results in laser-sintered SiSiC parts with mechanical properties similar to those of parts manufactured by means of conventional powder technology. These

are promising results which, given more time on research, might lead to even better convergence. With the combination of large-scale and micro-scale laser sintering in the described hybrid process, ceramic parts can be produced economically. With the use of other starting materials like hydroxyapatite and polyether ether ketone, laser sintering is also capable of producing bioactive and biocompatible parts suitable as implants based on complex, open porous structures.

The authors hope to have shown the broad variety of possible applications and the huge potential of additive manufacturing, as well as of selective laser sintering in particular, for the manufacture of complex-shaped, 3-dimensional parts that are comparable in their mechanical properties to conventionally produced components. The ever so important aspect of a short time-to-market factor, as another advantage of additive manufacturing, was amplified with the introduction of the possibility of an economic hybrid process.

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