

# Laser Micro Sintering of Ceramics - Reaction Models and Results

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## Abstract

Laser Micro Sintering is a modification of Selective Laser Sintering. Due to its specific features modeling of the process mechanism has become an issue of increasing interest. With elementary metals as feedstocks general qualitative explanations of the sintering phenomena are possible by considering the transitions of modification and aggregational stages and certain thermo-mechanical effects. In laser sintering of ceramics material specificity is more explicit. Next to temperature dependent absorption due to the electronic bandgap of almost all pure ceramics some of the materials cannot be liquefied reversibly under normal technical conditions. In some cases the transient liquid phase that is crucial for the required fast fusion of the powder material, especially during laser micro sintering but also during selective laser sintering generally, can only be obtained from dissociation products. Presently all laser sintered specimens from the investigated ceramic compounds need subsequent furnace sintering. Correspondingly, the principles of laser-material interaction derived from the observations with pure metals can be extended from metal to ceramics only to a very restricted extent. Results with Alumina/Silica, SiC and SiO as respective representatives of “oxide”, “non-oxide” and “sub-oxide” ceramic feedstocks are presented; qualitative process mechanisms are suggested.

Keywords: Rapid Prototyping & Manufacturing, Micro Technologies, Laser Micro Sintering, Sinter Mechanism, Alumina, Silicon Carbide, Silicon Monoxide.

## 1 Introduction

The principle of selective laser sintering (SLS) is the repeated coating and selective densification (sintering) of powders - in fewer cases also pasty materials - with the ends of generating a three-dimensional body layer by layer. The powder is swept across the lowered powder bed by a suitable tool, in the most case a doctor, leaving a thin layer of yet un-sintered powder on the top level of the bed. The sintering is achieved by scanning a laser beam across the entire admeasurements of the intended object's cross-section [Fig. 1]. Since its invention by Carl Deckard & colleagues [1] Selective Laser Sintering has been upgraded continuously to meet the requirements for the production of functional components [2].

### 1.1 Laser micro sintering - a step towards higher resolution

After the improvement of the resolution had come to a halt for a few years at about 100µm [3], in early 2003 the benchmark for the resolution of selective laser sintering was lowered considerably beneath the limits commercial SLS devices had been confined to [4] by the report of a functional tool made from tungsten powder. The new technique was named Laser Micro Sintering.

Further on, an increasing number of additional metal powders were processed successfully. The obtained structures showed a resolution of less than 30µm for overall resolution, of 20µm for ligaments and of 10µm for notches at aspect ratios of 12 and above, and presently a minimal roughness  $R_a$  of 1.5µm.

### 1.2 Laser micro sintering - the technique and the equipment

To obtain the required narrow focus for the high resolution initially the wavelength of 1064nm was chosen (Nd:YAG-laser in TEM<sub>00</sub> mode [4]); an additional reason for this choice was the commercially available variety of these lasers. Because of the feedstock's properties and reaction behavior q-switched pulses were applied [5,6]. Lately multimode radiation and other lasers with various wavelengths are used. In the typical laser micro sinter equipment the powder coating device with the sinter platform is enclosed in a vacuum tight casket. A window in the lid allows for incidence of the laser beam. In this arrangement processes can be conducted under a strictly

controlled atmosphere. Instead of by a doctor the powder is coated by a hollow vertical cylinder - loaded with a powder batch of around 10cm<sup>3</sup> - that is slid across the sinter platform.

## 2 Irradiation regime and sinter strategy

### 2.1 Thermodynamic effects of q-switched pulses during laser sintering

According to the prevalent hypothesis the interaction of q-switched pulses with powder materials [5,7] implies, next to heating and concomitant change of the aggregational states, also the transient exertion of a high pressure from an expanding plasma. During this miniature blast the eruption of the remaining overheated melt is shortly detained and the liquid entity is molded onto the underlying surface. Release of a limited amount of vapor and instantaneous solidification are the final steps of a “pulse event” and result in a flat dot of newly sintered material from each pulse. Rapid plasma expansion and vapor emanation are assumed to screen the remaining heated material from the surrounding atmosphere e.g. to prevent oxidation of the heated material during the crucial phase of fusion and solidification. There is evidence that under certain prerequisites laser micro sintering can be performed under normal atmosphere without corrosion [5,7,8]. This “reaction-shield effect” is very evident in the processing of metal powders but can also be observed during laser sintering of non-oxide ceramics, as shall be described later in this article.

An additional effect of a q-switched pulse is that within 10-100µs the main amount of its energy is transferred into a tightly restricted entity of the powder layer. Thus, by ideal pulse-wise sintering collateral fusion of the powder outside the laser spot through heat conduction is mostly avoided.

### 2.2 Laser wavelength and absorption by solid bodies

As mentioned above (1.2) for several reasons Nd:YAG-lasers with a wavelength of 1064 nm and a corresponding photon energy of 1.17 eV. were chosen for laser micro sintering. Electromagnetic excitation of inorganic solids (not considering “molecular crystals”) by near infrared radiation is only absorbed by electrons. In dielectrics the orbitals for the potentially excitable electrons are separated by the “band-gap” into the “valence band” and the “conduction” band. In the ground state the valence band is completely occupied and the conduction band completely empty. The electron configurations of many dielectrics at room temperature approximate this state. Therefore, the only possible excitation is from the valence band into the conduction band. The required energy has to exceed the bandgap. This accounts for the poor absorption of near infrared radiation at room temperature by many dielectrics. Strictly speaking the only considerable near infrared absorption under these conditions should be via non-linear effects that can be achieved with highly intensive radiation. Once the dielectric is excited, rise of temperature and concomitant increase in equilibrium electron occupation of the conduction band are induced. The Fermi-Equation (1) describes the occupation probability of the conduction band as a function of  $E$  ( $E$  being at least as large as the lowest state in the conduction band;  $E_F$  denotes the Fermi energy).

$$f(E) = \frac{1}{1 + e^{\frac{E-E_F}{kT}}} \quad (1)$$

The resulting marked increase of the absorption coefficient for all photons with lower energy than the band-gap value is approximately proportional to the rise in the conduction band occupation. The avalanche-like heating of

the material due to this behavior bears the danger of disintegration if irradiation is not cut short at a certain degree of excitation. This is almost impossible with an irregularly packed material like the powder layers in laser micro sintering.

### **2.3 Photonic effects of q-switched pulses during laser sintering**

Due to the high pulse-peak powers the probability of multi-photon excitation (excitation by nonlinear effects) of the irradiated material is considerably higher than during the respective processing with a laser that is operated in a continuous wave mode. This feature is crucial for the interaction of the radiation with materials that do not offer the chance for direct absorption as has been explained in the previous paragraph. The limited duration of the irradiation due to the narrow pulse width in the order of  $10^{-8}$ - $10^{-7}$ s makes it possible to suppress the overheating of the material that is impendent because of the instantaneously launching and self-accelerating direct absorption after initial excitation. An additional self-limitation due to the enhanced plasma generation at high peak powers and the concomitant radiation-shield effect might also be discussed.

### **2.4 Essential differences in the sinter strategy for metals and ceramics**

The generation of a defined body by selective laser sintering relies on the transient – at least partial – conversion of the powder material into liquid phase. The liquid phase wets and fuses adjacent and embedded solids. Terminatory solidification yields the sinter volume supplement of the respective layer.

Due to the respective properties of metals these consecutive processes can usually be realized within the inherently short reaction time of a pulse event, which means that by each q-switched laser an isolated persisting sinter-spot is generated. The prerequisites for this are high absorption of the radiation, a sufficiently wide temperature range for the liquid phase, and a low viscosity of the melt.

Direct laser sintering of ceramics is less straightforward. Besides the already described absorption difficulties that arise from the electronic configuration of dielectrics, the melt phase of the ceramic compounds decomposes at a certain temperature. Some of the materials cannot be reversibly liquefied at all under technically feasible conditions. Pulse-by-pulse sintering usually is not successful with ceramics so that special sinter strategies have to be applied that cause a simultaneous melting and fusion of larger entities. This is achieved by grouping the pulses and placing each group of consecutive pulses very close to each other in an approximately circular dot so that the corresponding melt “blob” does not cool as rapidly as a sinter spot generated by a single pulse. This offers the material prolonged time for fusion. An explanation for this necessity might be found in the comparably higher viscosity of a ceramic melt phase, especially when it has considerable ionic character.

## **3 Laser micro sintering results with oxide, nonoxide, and suboxide ceramic powders**

### **3.1 Results of laser micro sintering with alumina ceramics**

Alumina has a melt phase that is stable between 2050°C and 2980°C and thus fulfills one of the crucial requirements for laser sintering. Because of its band gap, however, problems arose initially with continuous Nd:YAG-laser radiation ( $\lambda = 1064\text{nm}$ ). Due to the mentioned avalanche effect it was impossible to sufficiently control the uptake of continuous laser energy by the material. When working with continuous radiation collateral heating of the powder surrounding the actual spot of incidence had to be taken into account. Collateral heating generates something like a thermal prow wave on the powder layer in front of the moving laser spot. Under these

conditions it is possible to sinter the material with a continuous laser beam at a power, low enough to avoid excessive decomposition of the material, as there is always a certain degree of thermal activation of the powder shortly before the arrival of the spot center. As the results with an alumina/silica blend showed [Fig. 1a], the large extent of collateral heating, nevertheless, leads to a deterioration in the resolution of selective laser sintering. Sufficient confinement of fusion to the irradiated material was only achieved with q-switched laser pulses [Fig. 1b].

Following an optimization of the powder coating routine of the sinter strategy as well as a choice of an appropriate laser radiation source, bodies were obtained from an alumina/silica blend with an accuracy between 50 $\mu$ m and 80 $\mu$ m. The laser sintered product has a vitreous appearance and a poor pressure strength; high pressure strength and opacity were obtained only after an additional furnace sintering step [2] during which a shrinkage of 0.7% occurs. FEM cross section views suggested a low content of mullite after laser sintering and a considerably higher content after the subsequent furnace process [9]. Meanwhile XRD measurements confirmed this assumption [Figs. 3a,b; 4a,b]. The conclusion from this observation is now that after the comparably rapid laser sintering process that takes place within seconds, the rather heterogeneous crystalline material is bonded prevalently via – relatively brittle - amorphous phases [Figs. 3a, 4a]. The required time for diffusion and recrystallization, however, ranges within hours. This time is provided during furnace sintering after which a firm solid body with an opaque appearance is obtained. The probes displayed in Fig. 5 demonstrate the state of the art.

### 3.2 Results of laser micro sintering with non-oxide ceramics

Silicon carbide cannot be reversibly transferred into liquid state at normal pressure conditions. It decomposes around 2800-3000°C. The smallest possible bandgap of SiC is 2.4eV (in the 3C-SiC modification) which is yet too large for the quantum energy of 1.17eV delivered by the 1064nm radiation of an Nd:YAG-laser. This is the reason why the successes in laser micro sintering of SiC were obtained with a silicon blended powder material, SiSiC [6]. As silicon has a bandgap of 1.1eV, absorption of the NIR pulse by silicon is considered the initiation of the sinter process. It is also silicon that yields the melt phase, at normal pressure in the temperature range 1420-3270°C, which is crucial for laser sintering. It is assumed that part of the liquefied silicon in the irradiated powder volume is evaporated and thrust off the surface as a miniature rapidly expanding gas or plasma bulb (see 2.1). By this event the rest of the melt is forced towards the surface or in between the powder grains in the direct vicinity of the impact. The resulting fusion of undissociated SiC-grains suffices for the selective generation of a, though brittle, however dimensionally stable solid body. Accordingly, opposed to recent assumptions [5,6], the build-up of solidified material rather takes place on the rim than in the center of the laser spot. In Fig. 6 it is visible that each of the lines that consist of a row of cut-ins from the laser pulses is confined by walls of solidified material. The cut-ins vary in depth and size as the powder layers usually are not ideally homogeneous. The observed lack of oxidation - although the process is conducted under normal atmosphere - corroborates the assumption of rapid plasma expansion with the concomitant reaction shield effect.

Meanwhile there is evidence that principally the same sinter mechanism takes place during laser micro sintering of unblended SiC powder. The only difference, compared to the process with Si blended powder, is that the absorbant, the silicon, has to be produced by dissociation of SiC during the sintering of the respective previous layer or layers. One plausible explanation is that the laser radiation is absorbed by the content of silicon and maybe some carbon in the already solidified material. Thereupon the adjacent unsintered powder is thermally

activated for absorption and decomposition, the latter one setting free the silicon required for the fusion of undissociated SiC grains. There might as well occur partial decomposition of the already solidified - better absorbing – material, yielding the binder for the interconnection of yet unsintered powder grains and providing a thermally activated site for the fixation of the newly sintered material onto the body.

In any case, the underlying sintered body seems to have a crucial role in the progress of solidification: On the rim of the sintered layers usually a tarnish of partially solidified material can be observed [Figs. 7, 8] originating from silicon blown into the powder bed. For want of sufficient contact with activated sintered material, however, it remains detachable. In the horizontal CT-view of Fig. 8 the tarnish is visible as a seam outside the actual outline (marked by a yellow dotted line) of the body. Because of the same reason typical defects can be observed in laser micro sintered SiC bodies [Figs. 8, 9]. Due to randomly occurring gaps in the uppermost sintered level the following powder coating becomes locally too thick to allow for activation of the already sintered surface. Either voids result from this or insufficient attachment of the next layer leads eventually to delamination [Fig. 9]. Since laser micro sintering of the unblended SiC, compared to SiSiC, requires a stricter control, a regime with continuous wave radiation under an inert shield gas was applied until recently. Improvement of the powder packing reproducibility finally made it possible to process the unblended powder under normal atmosphere [Fig. 10]. After laser sintering the bodies are still very brittle. Due to the open porosity the specimens can be infiltrated with silicon whereby they acquire a pressure strength of 1000MPa and above.

### 3.3 Laser reaction sintering with silicon monoxide

#### 3.3.1 Reactions of silicon monoxide upon laser irradiation

Preliminary experiments had shown that silicon monoxide (SiO) absorbs sufficiently radiation of the NIR spectral range. This was considered a chance for selective sintering of ceramic-like micro-bodies consisting of SiO<sub>2</sub> [10].

There are reportedly two known modifications of solid SiO, the graphite-like type and another modification that is usually referred to as amorphous SiO. The below described observations were obtained from experiments with the amorphous modification. The material has a liquid phase between 1700°C and 3200°C [11,12]. There exists a defined species SiO in the monomolecular gas phase. In the solid state stoichiometric distribution of the elements on the atomic scale can only be found in very thin films [12,13]. Soon after deposition of more voluminous bodies gradual disproportioning starts, resulting in phases of silica and ‘amorphous clusters of silicon’. Under an annealing temperature of 900°C disproportioning of 80% is reached after ten seconds, the following growth of silicon crystals with diameters around 5nm affords reaction times in the order of hours [14,15,16].

Originally all of the below listed reactions were regarded potentially suitable for sintering:



Considering the reported complexity of the material it becomes evident that a straightforward sinter process is not to be expected. Although the oxidation reaction would be the most favorable for a generative technique, it is not to be expected at full extent because of the very limited potential reaction time in laser micro sintering during

which sufficiently heated material can be accessed by oxygen from the environment. Disproportioning does not necessarily involve the rearrangement or fusion of crystals as the reaction. Simple melting and fusion of unreacted SiO cannot realistically be presumed either, because of the poor possibilities for temperature control during laser processing of sub-micrometer powder material.

### 3.3.2 Results of laser reaction sintering with silicon monoxide

Consequently, due to the multiplicity of the reactions and the possible intermediates, the laser sintered bodies consist of a not yet clearly defined composite from several oxidation and disproportionation stages of the original powder. The composition and consistency varies according to the laser parameters and the scanning regime. As already mentioned as typical for laser sintering of ceramics (chapter 2.4) sintering with singular q-switched laser pulses is not suitable. Considerable dimensional stability is achieved only when a sinter strategy is applied by which the sinter layer is generated via the fusion of small partitions of the powder coating [Fig. 11a]. Pressure strength is increased by subsequent oven sintering which presently yields a maximum of 300 MPa, the appearance of the body changes from vitreous to opaque during this step [Fig. 11b].

If high fluences are chosen that yield optimum oxidation (estimated from the brightness and opacity of the product) the as-laser-sintered compound is well interconnected, also high accuracy and resolution are achieved, [Fig. 11c] but the relative density of the body is very low. With less fluence the as-laser-sintered body is very brittle although the relative density is much higher and can be improved up to 70% after subsequent oven sintering. The resulting micro parts show still highly resolved details [Fig. 11d]. Presently, however, these properties are obtained at the expense of the fidelity due to the considerable shrinkage (in the order of 10%) during the oven process.

## 4 Summary

Already the laser micro sintering process with metals relies on the synergism of several simultaneous effects and processes initiated by high intensity laser radiation with pulse times of 10-100µm. The complexity exceeds by far a mere succession of fusion and solidification. When the technology is extended to non elementary compounds, dissociation of the material has to be taken into account. Especially with covalent compounds problems arise from the lack of a liquid phase. Moreover, most ceramics are dielectrics with a noticeable temperature dependence of near infrared radiation absorption. This complicates the control of a laser process with these materials especially when their consistency is as inhomogeneous as it is the case in a powder layer. Reaction models are necessary for the principal understanding of the involved processes.

The multiplicity of the intermediates and the decomposition products as well as the slow rearrangement of the material to compound with sufficient firmness leads to a strategy by which a body with dimensional stability is generated by the laser process with the need of posterior treatment – furnace sintering and/or infiltration – to obtain appropriate material properties for functional applicability.

## Acknowledgement

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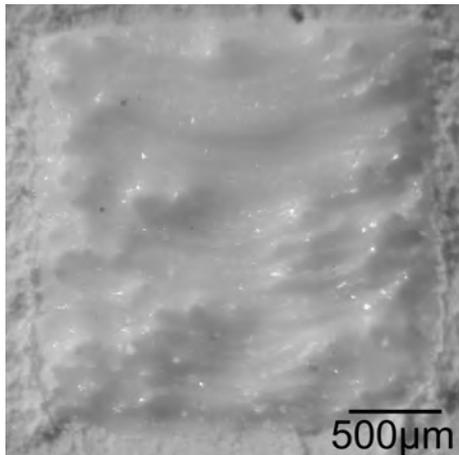
#03X0033B “KONAMI” (WING). Part of the presented results belong to a project #9649/1464 “Verfahren zur schnellen Erzeugung von keramischen Zahn-Inlays” supported by the European fund for regional development (EFRE) 2000-2006 in the course of technology promotion and by funds of the “Freistaat Sachsen”.

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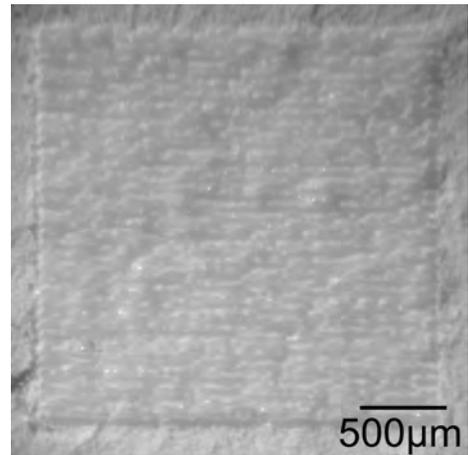
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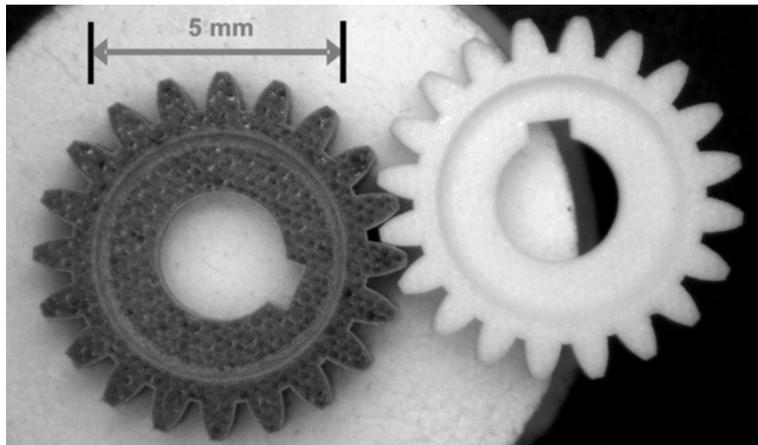
**Figures:**



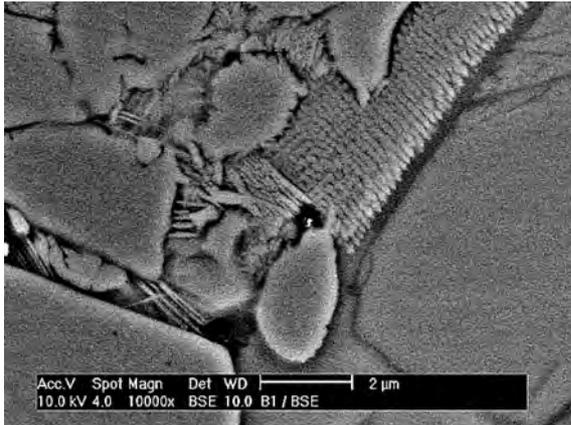
**Fig. 1a:** Sinter result with continuous NIR radiation applied at alumina/silica powder.



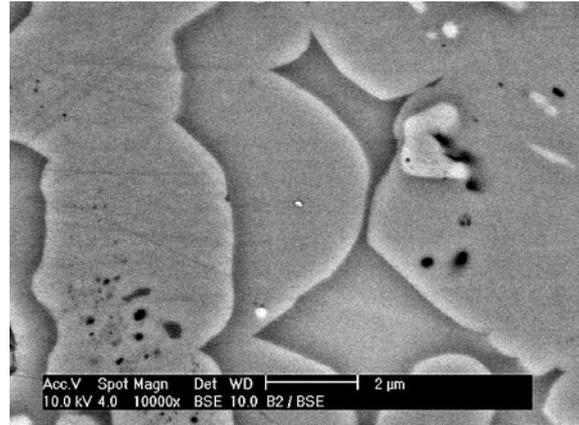
**Fig. 1b:** High resolution by selective irradiation with q-switched laser pulses.



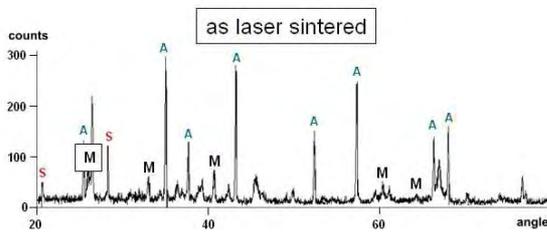
**Fig. 2:** Functional components from an oxide ceramic powder as-laser-sintered (left) and after additional oven sintering (right).



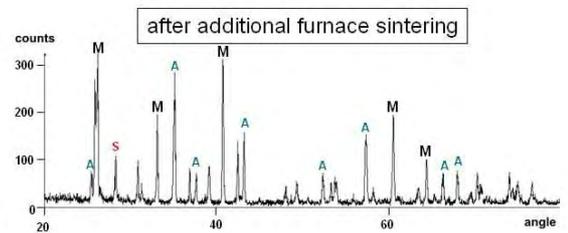
**Fig. 3a:** Cross section view as laser sintered.



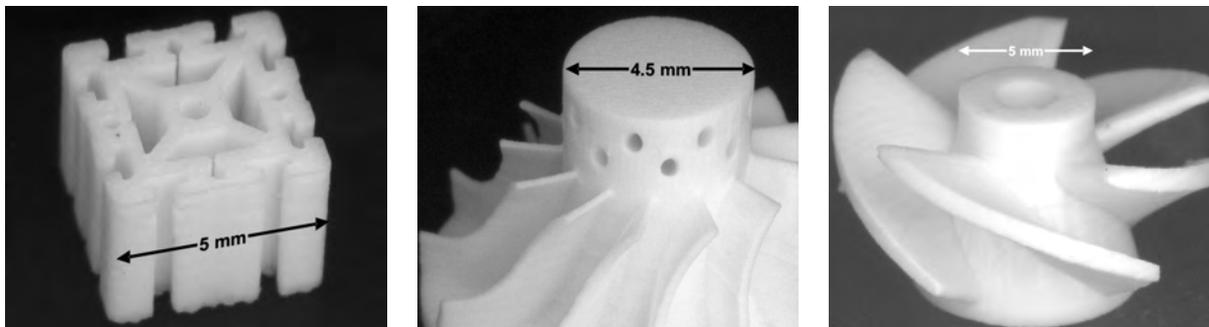
**Fig. 3b:** Cross section after furnace process.



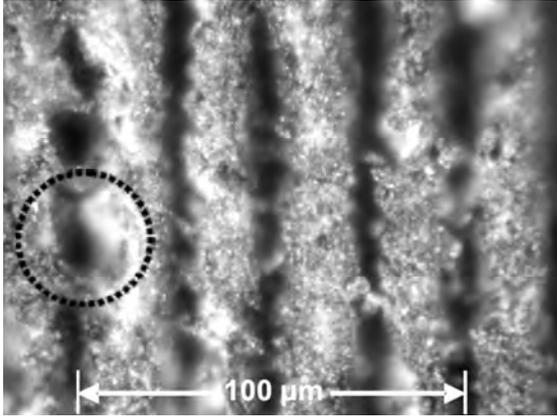
**Fig. 4a:** XRD mullite signals (M) after laser sintering. S  $\triangleq$  SiO<sub>2</sub>, A  $\triangleq$  Al<sub>2</sub>O<sub>3</sub>.



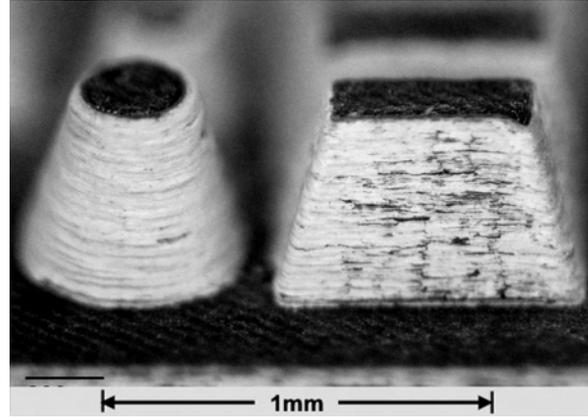
**Fig. 4b:** XRD mullite signals (M) after furnace process. S  $\triangleq$  SiO<sub>2</sub>, A  $\triangleq$  Al<sub>2</sub>O<sub>3</sub>.



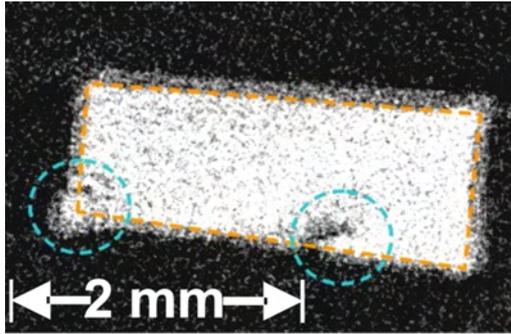
**Fig. 5:** Laser micro sintered parts from alumina/silica after subsequent furnace sintering.



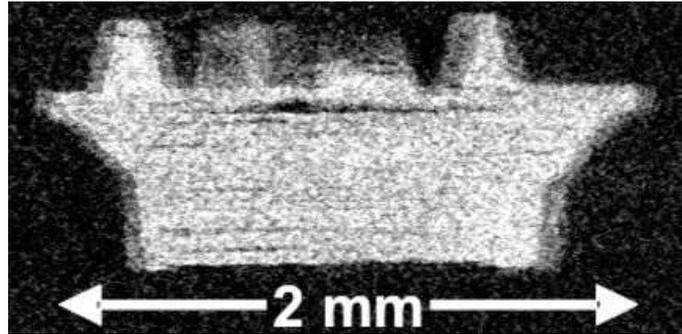
**Fig. 6:** Horizontal surface of laser sintered body from SiC. The furrows cut by line scans of pulsed radiation are lined by solidified crests.



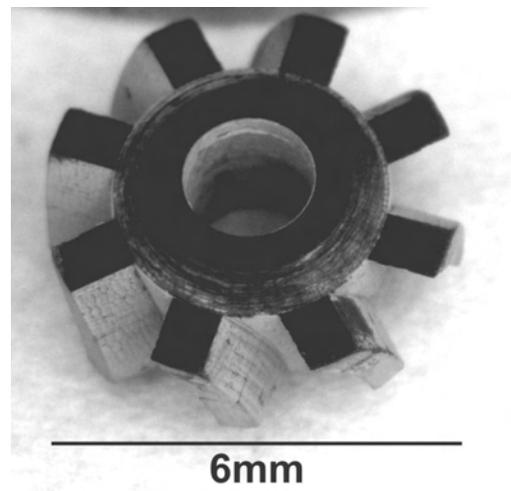
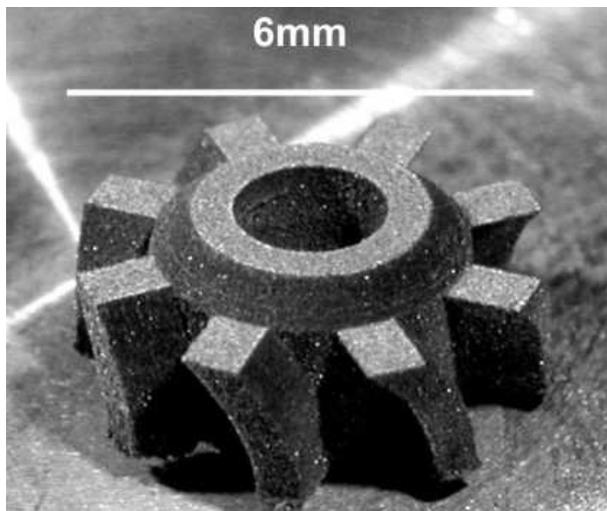
**Fig. 7:** The white tarnish on the vertical surfaces consists of condensed and oxidized material from the plasma torch.



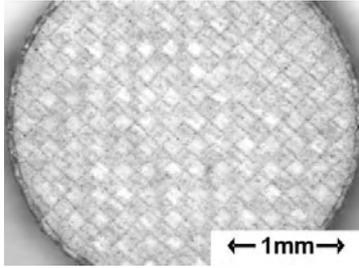
**Fig. 8:** CT-view of a horizontal cross section. The grey seam outside the body outline is the white tarnish. The dotted circles mark insufficiently sintered spots.



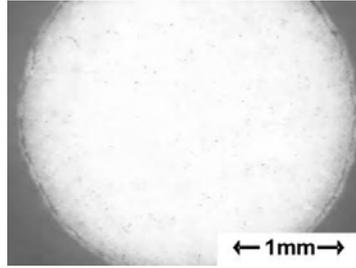
**Fig. 9:** Random gaps in the sinter layers are the cause of voids and delamination.



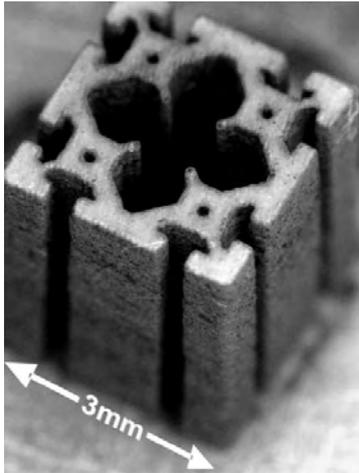
**Fig. 10:** Because of the similar sinter mechanisms both SiSiC (left) and SiC (right) can be processed under normal atmosphere. The two specimens have not been infiltrated yet.



**[Fig. 11a]:** Surface of an as laser sintered specimen from SiO.



**[Fig. 11b]:** Surface of the probe after subsequent oven sintering.



**[Fig. 11c]:** Object with high resolution and low density, laser sintered with high fluence.



**[Fig. 11d]:** Specimen that had been laser sintered with low fluence after subsequent oven sintering.