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# Laser micro sintering – a new method to generate metal and ceramic parts of high resolution with sub-micrometer powder

H. Exner, M. Horn, A. Streek, F. Ullmann, L. Hartwig, P. Regenfuß and R. Ebert

University of Applied Sciences Mittweida, Laserinstitut Mittelsachsen e.V., Germany

Laser micro sintering (LMS) was developed by the research group at University of Applied Sciences Mittweida and the associated Laserinstitut Mittelsachsen e.V. as the result of research started in 2001 with a project on the possibility of generating parts by selective laser sintering (SLS) with improved resolution. For the successful generation of solid bodies from various metal powders the technology uses essentially sub-micrometer powders, a cylindrical coating blade and a q-switched solid state laser. The resolution and the surface roughness are by more than one order of magnitude better than achieved by previous selective laser sinter technologies. Presently the technology shows advancements in selective laser sintering of highly resolved specimens of densely sintered  $\text{Al}_2\text{O}_3$  and SiC ceramics too.

This paper reports the process mechanism of LMS and its principal differences compared to other SLS methods. A variety of laser micro sintered parts from different metals and the newest results in laser micro sintering of ceramic parts are presented. Material specific behaviour in laser micro sintering is discussed. Also the ability of the method will be shown to generate parts of layer wise different materials (laminated sintering) in one sintering machine.

*Keywords:* laser micro sintering; selective laser sintering; sub-micrometer powders; metals; tungsten; alumina; SiC

## 1. Introduction

Selective laser micro sintering (SLS), which was developed by Laserinstitut Mittelsachsen e.V. (Ebert and Exner 1999) is used for generation of micro parts. Tungsten and tungsten alloys (especially tungsten-aluminium) were employed as the first usable metal powders for laser micro sintering (LMS) (Regenfuss *et al.* 2003, Exner *et al.* 2003). Meanwhile many new materials were investigated (Regenfuss *et al.* 2005).

In the year 2004 the sintering of ceramic powders became a new field of investigation (Exner *et al.* 2005). Two different material groups for sintering ceramics were defined: oxide ceramics and non oxide ceramics. Besides this new investigations of sintering metals and process improvements were also progressed.

Many effects that occur during LMS are already investigated. Nevertheless process specific improvements can still be achieved.

However, for industrial application in many cases materials different from the already investigated are required. Meanwhile numerous powders of various materials and grain sizes have been employed for LMS, some of which allowed a considerably simpler process regime. Thus for example with certain materials laser sintering under normal atmosphere was realised.

## 2. Laser micro sintering of metal

Until recently, only metal powders with homogenous grain sizes were applied. They were usually coated with a sharp

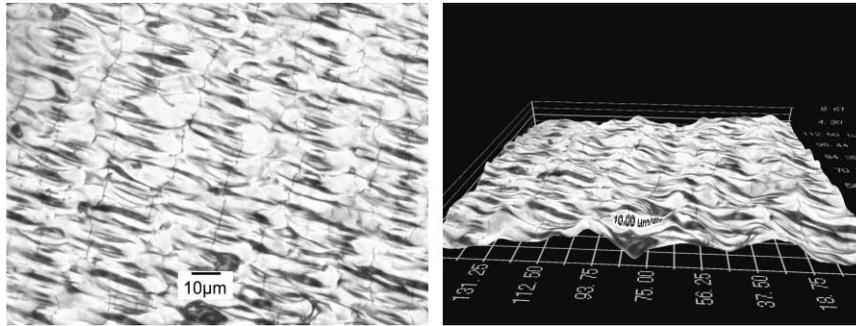


Figure 1. Left: surface view of the almost perfectly molten metal phase of a sintered body consisting of molybdenum. Right: 3D view of the surface.

blade to optimum quality of the powder layer for micro sintering. By the employment of a new rake that exerts pressure on the layer powders with a wider spectrum of grain size can be processed now. By using this new kind of rake and powder with a wider grain size spectrum also the density of the powder bed is increased and the density of the resulting micro parts is raised. If higher densities can be achieved the heating of the material will become more controllable and a more stable melting pool of liquid metal can be achieved because of the more constant dissipation of the absorbed laser energy into the material. The resulting specimens seem to consist of an almost perfectly molten metal phase without the spongy structure usually obtained by the intensive laser pulse treatment (figure 1).

A new problem can be observed (figure 1 left). It is commonly known that in densely laser sintered bodies of hard material the thermally induced stress causes cracks due to lack of relaxation possibilities. (figure 1 right) shows the 3-dimensional (3D) view of the surface. The maximum roughness  $R_z$  has a very good size of lower than  $5 \mu\text{m}$ .

The resulting high densities are also shown in the cross-section view of a generated specimen (figure 2). In the left corner of the upper part of the picture one can see a model of the total geometry of the sintered body. Only on the fringe, there is a lower density due to the additional shearing forces of the coating blade which result in an inhomogeneous powder layer. The lower part of the picture shows a cross section of the high density sub area and the low porosity of the sintered body.

As a result of the more homogenous powder coating and the higher density, specimens could be generated with thin walls of only  $40 \mu\text{m}$  and with a high aspect ratio of 1:25 (figure 3).

Newer investigations aim at the laminated sintering of two different materials. One of the processed material combinations is molybdenum with copper, another one silver-copper. In the case of Mo-Cu the necessary parameters for the copper powders were determined and compared to the parameters of molybdenum. The difference between the required parameters was too big to sinter

them together directly. Thus parameters for intermediate zone (transition zone) have to be found.

A special problem is the changeover from molybdenum to a copper layer, owing to the high reflectivity for the employed laser wavelength of  $1064 \text{ nm}$  for Cu. This high reflectivity yields a weak vertical interconnection due to the insufficient penetration of the covering copper layer. And a

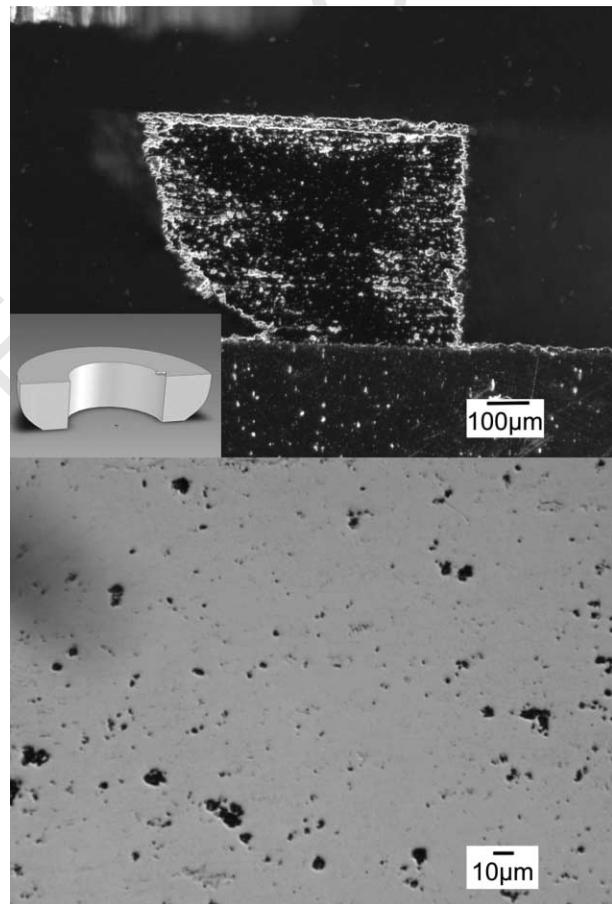


Figure 2. Upper part: cross-section view of a molybdenum specimen. Lower part- magnification of the cross-section of the high density sub area; a low porosity is seen.

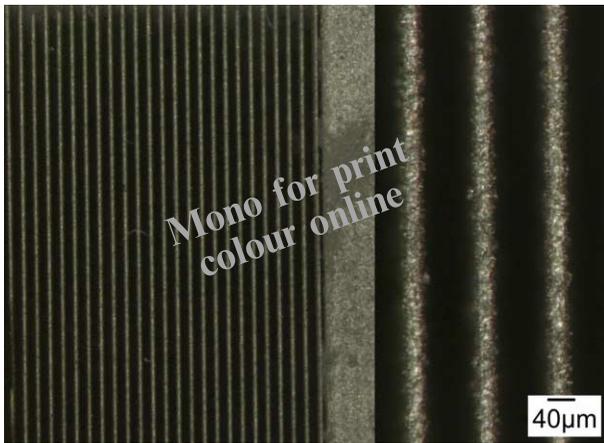


Figure 3. Left:  $40\ \mu\text{m} \times 1\ \text{mm}$  walls consisting of molybdenum. Right: magnification of the walls.

weak interconnection results in a crack between the materials. An important effect in this connection is the so called raising defect. This effect produces a trailing of the actually sintered layer behind the coated layer.

By the variation of the pulse duration and average laser power, this tracing can be minimised to the interconnecting zone between molybdenum and copper. Thus the crack-free combining of the two materials is possible (figure 4).

### 3. Laser micro sintering of oxide ceramics (alumina)

A specific challenge for LMS is the processing of non metallic materials. The necessary high intensities (Regenfuss *et al.* 2006) can be economically achieved only by using near infrared lasers. However using this group of lasers the material specific absorption has to be observed. Especially oxide ceramic materials are almost completely transparent in the near infrared the usual wavelength for q-switched solid state lasers.

Starting in 2004 the LMS technique until this time only was applied to generate solid bodies from metals. In the middle of 2005 first successfully results could be achieved to generate oxide ceramics bodies (Exner *et al.* 2005). But to fulfil the requirements of laser sintering of ceramics it was necessary to develop new processing technologies and parameters (Exner *et al.* 2006):

- the grain size of the powder has to be in the sub-micrometer range to reach a requested resolution of  $40\ \mu\text{m}$  for this technique;
- the absorption coefficient of the laser wavelength has to be high enough to transfer the laser energy into the material;
- the generated sinter layers have to have a defined mean density of the material;

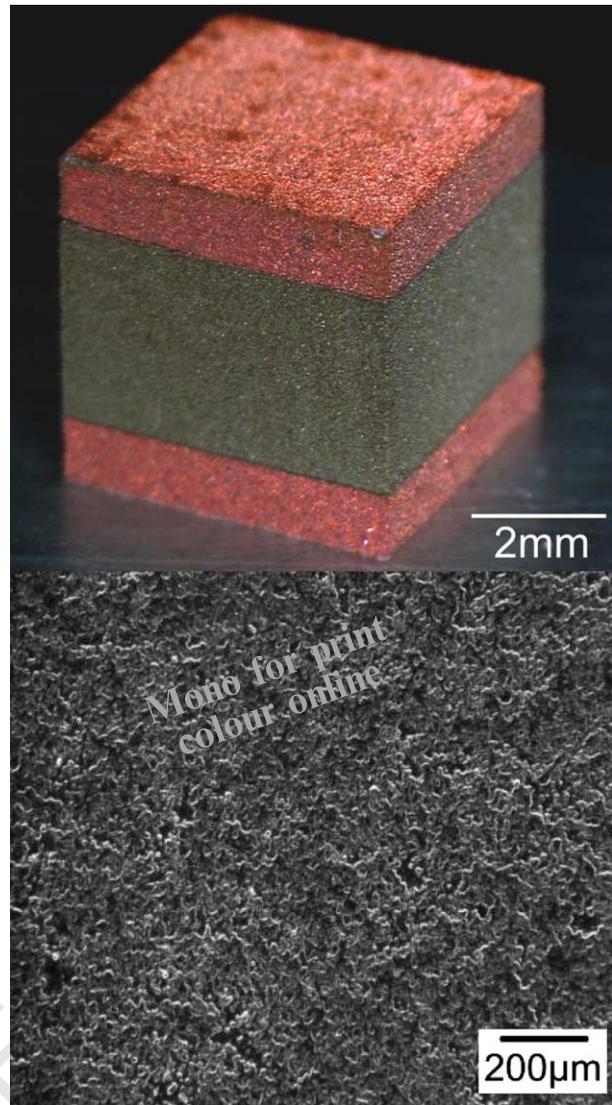


Figure 4. Upper part: sintered laminated cube of copper–molybdenum–copper. Lower part: magnification of the copper surface.

- the processed material has to have a certain composition of glass and crystalline fraction to generate a satisfactory sinter quality.

To solve these and other problems the initial experiments started with pre-pressed pills of powder. To show the influence of the of powder composition figure 5 shows an example of a processed feldspar powder that contains too much silica.

The result is a voluminous body without a structured outline of glass-like consistence. The solid has also some small blisters of gas inside. The material is probably decomposed during the laser process. As one can see in the lower area of the picture the transformation of material

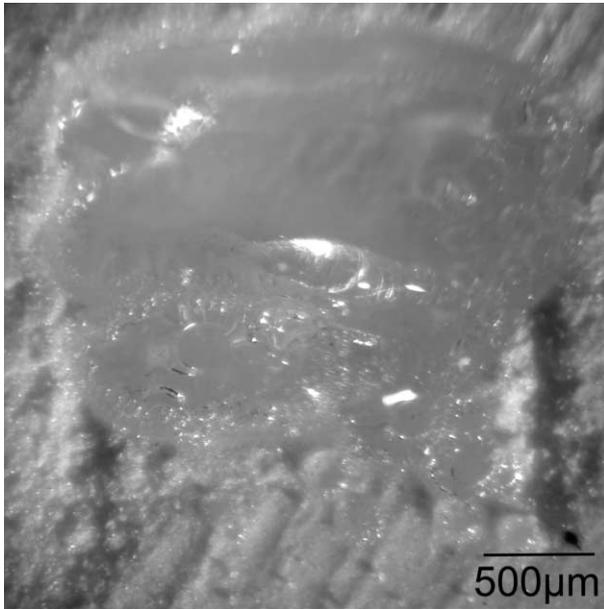


Figure 5. Surface of a laser sintered section of compressed feldspar powder, processed with 1064 nm cw-radiation.

started not at the beginning of the laser beam interaction but later.

Owing to the dependence of the absorption coefficient of the material on the temperature itself one generates an avalanche process ending in a non defined starting of melting and hence a non-defined geometry of the layer.

Consequently, modification of the material was necessary with a reduced amount of glass-forming components. A mixture of two components: alumina and silica was optimised to obtain a product with more ceramic appearance and a somewhat higher degree of shape fidelity. A regime was developed reduce the risk of decomposition at the cost of outline resolution, which was still very low (figure 6).

But finally only by the use of a q-switched laser beam with its much intense short pulses (ns) satisfactory results could be obtained for the sintering of a thin layer with an exact and sharp boundary (figure 7).

By scanning the surface one can generate a closed surface layer of a sintered ceramic volume. This was the premise for a layer wise liquid phase sintering of a body and the better control of the described laser sintering process of oxide ceramics.

If we calculate the intensity of the laser spot it is a factor of several  $10^4$  higher for the case of q-switched pulses against continuous wave interaction. Thus a part of the ceramic powder evaporates and causes very high pressure by the rejecting forces of the vapour – the second necessity besides temperature to generate a sintered layer.

After a development of about two years we achieved laser micro sintered ceramic bodies with a resolution up to 40 μm

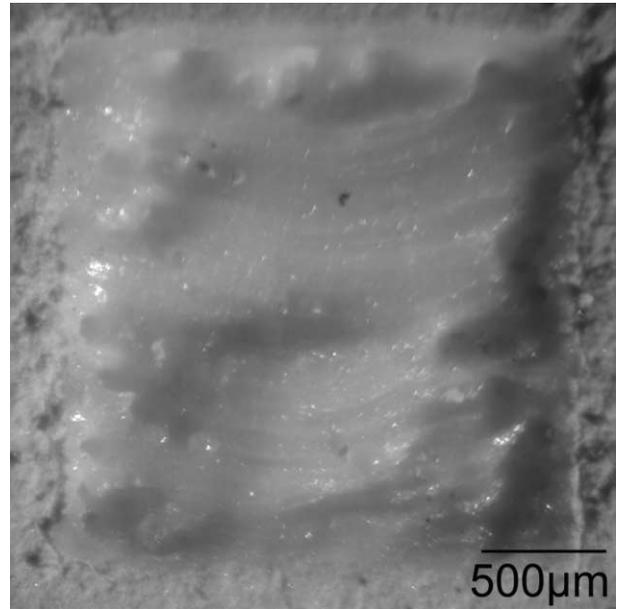


Figure 6. Wavy surface of compressed synthetic ceramic powder, sintered with 1064 nm cw-radiation.

and an average surface roughness  $R_a$  of about 5 μm by the improvement of the sinter strategy and the optimisation of the powder composition. The maximum relative density depending on the dimension of the sinter layers is about 98%.

Furthermore, the appearance and the structure of the ceramic parts can be improved by a thermal treatment after laser sintering. The shrinking rate of the ceramic bodies was

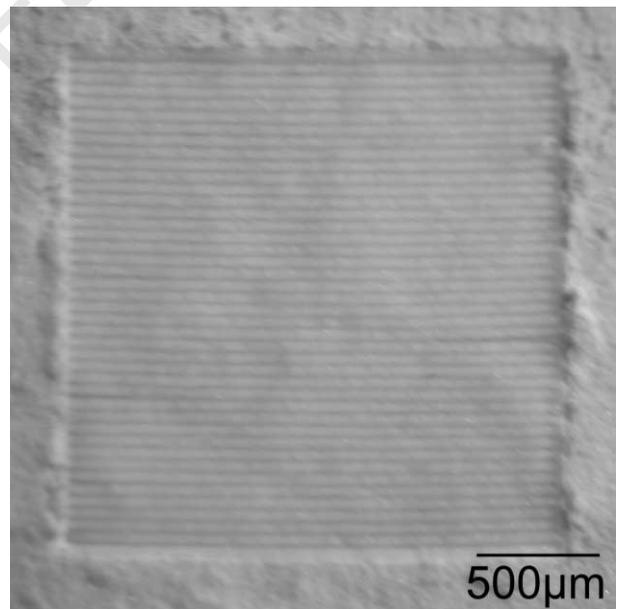


Figure 7. Surface of compressed ceramic powder, sintered with 532 nm q-switched radiation.

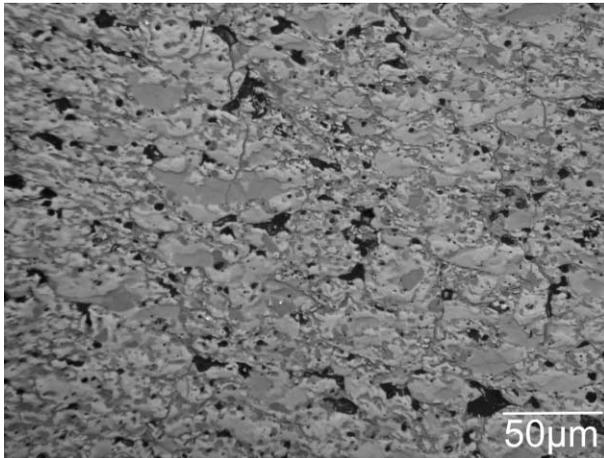


Figure 8. Cross-section of a laser micro sintered oxide ceramic body.

tested on  $8 \times 8 \times 1 \text{ mm}^3$  parts and was measured as significantly smaller than 1%. The reason is that the powder material for the direct laser sintering process does not contain any binder that leaves pores after its removal.

figure 8 is a cross-section view of a direct laser sintered material. It reveals many small pores and micro cracks inside the sintered body. But after thermal treatment the cracks seem to be annealed. The number of pores decreases too. Some bigger pores are left (figure 9).

In both pictures we find three different tones beside the pores. A modification of the material structure can be observed. During the sintering process a new material must be accumulated maybe a mullite ceramic.

In comparison of both cross sections views one can find many small areas of material components before this procedure and bigger regions of components after it.

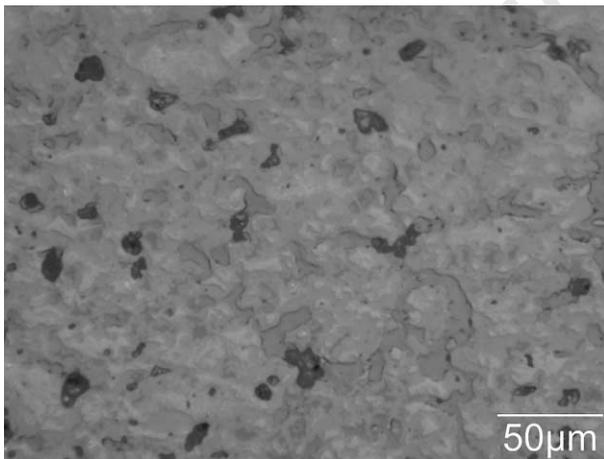


Figure 9. Cross-section of a laser sintered oxide ceramic body after thermal treatment.

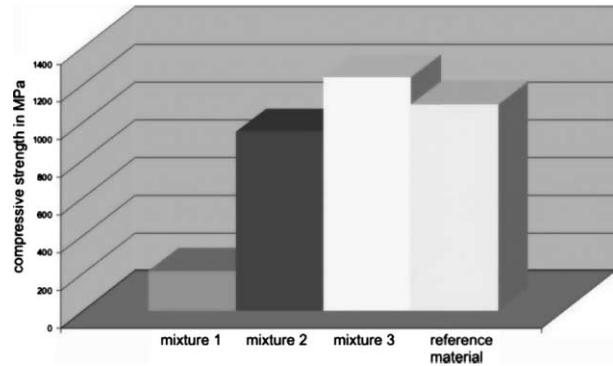


Figure 10. Achieved compressive strength of different composed materials in comparison to our reference material.

If the measured compressive and bending strength before and after annealing are compared, it turns out, that these properties also have improved also. figure 10 shows the results of compressive strength depending on different mixtures of alumina and silica in comparison of a reference ceramic material sintered with conventional methods. By using several building rates ( $\text{mm}^3/\text{h}$ ) much better results of bending strength are achieved than within our reference material (figure 11).

To compare the strength of the laser sintered ceramics with that of the reference material bending-strength measurement were conducted with 30 probes according to the four-point bending method and achieved the Weibull distribution shown in figure 12 was obtained. For the laser sintered ceramic parts a much more statistical spread was observed than for the reference material. The conventionally produced ceramic has a steeper rise that means a lower spread in the testing results so it is easier to estimate the damage of the components (figure 12).

But on the other side the laser sintered ceramic has a higher balance point and the minimum result is still much better than conventionally produced ceramic. By improvement of the sintering conditions a similar curve progression

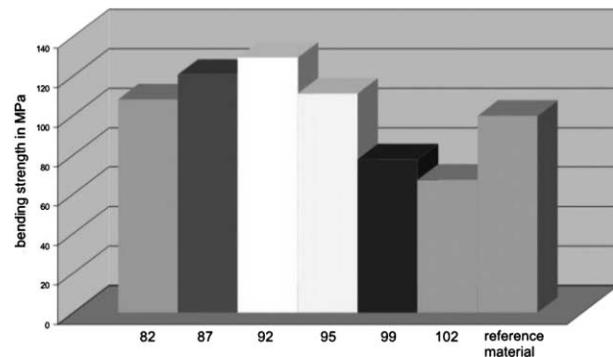


Figure 11. Achieved bending strength by different building rates ( $\text{mm}^3/\text{h}$ ) in comparison to our reference material.

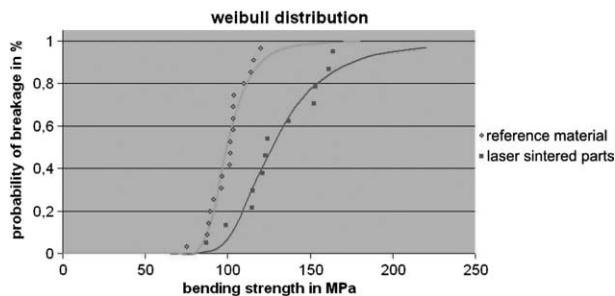


Figure 12. Weibull distribution of laser sintered ceramic in comparison to our reference material.

like the reference ceramic but with higher minimum cracking probability can be expected.

The examples in the figures below demonstrate the accuracy of this technology and the high geometric resolution. figure 13 shows a compilation of laser sintered oxide ceramic parts. The hollow balls for example demonstrate very impressively the possibility to generate undercut.

The used powder is a mixture of alumina and silica. The ceramic parts are all composed by 2.5  $\mu\text{m}$  sinter layers and processed by our q-switched laser source. Some of these parts were thermally treated to achieve much better mechanical and optical properties like compressive and bending strength and different white tones too.

#### 4. Laser micro sintering of sub- and non-oxide ceramics

##### 4.1 Silicon monoxide

As mentioned above, especially oxide ceramic materials are almost completely transparent in the near infrared. In

comparison silicon monoxide has a good absorption for the used wavelength of 1064 nm and can be easily oxidised under environmental atmosphere to silica. Thus a new modification of LMS has to be investigated called 'laser reaction sintering'. In this modification the material after the laser treatment is different from the material at the beginning of sintering. Thus, during the sinter process the material is chemically modified. As first material for laser reaction sintering of silicon monoxide was chosen.

As the first results show, silicon monoxide can be sintered easily by using standard parameters known from sintering metals. Also it can be observed, that the variation of the pulse duration results in two kinds of phases of  $\text{SiO}_2$ . At low intensities a highly amorphous phase is sintered; at higher intensities at the same fluency more crystalline sub areas are resulting (figure 14). Also the bending strength depends on this kind of phase. This fact has to be more investigated in further activities. By using the present empiric parameters for the reactive sintering complex specimens can also be generated (figure 15).

##### 4.2 Silicon carbide

The results presented in figure 16 were been gained from experiments with silicon enriched SiC ('SiSiC') containing an additional 8% carbon and with technically pure SiC. The additional content of carbon in the silicon blended powder was supposed to yield information on the chance of secondary SiC formation. Anticipating a possibly poorer temperature and wear resistance of the products assays were also conducted with technical grade SiC.

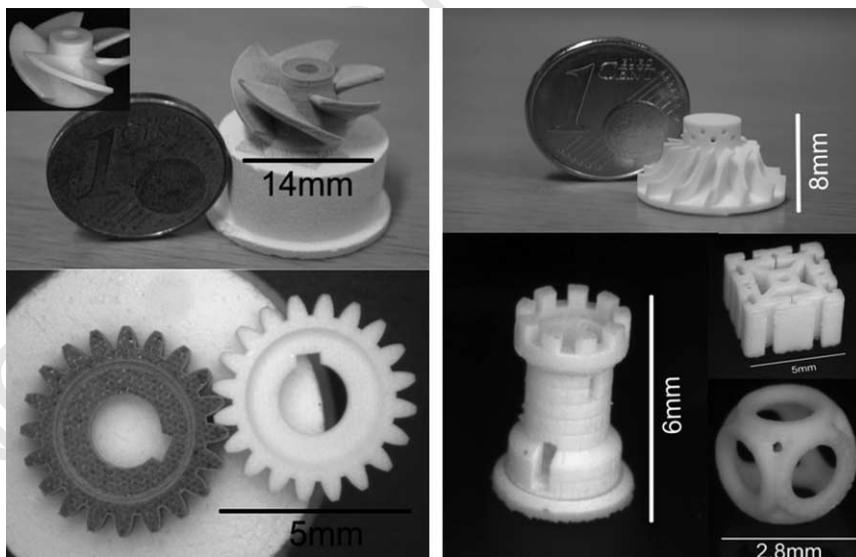


Figure 13. Some examples of laser micro sintered oxide ceramic parts (Ebert et al. 2006).

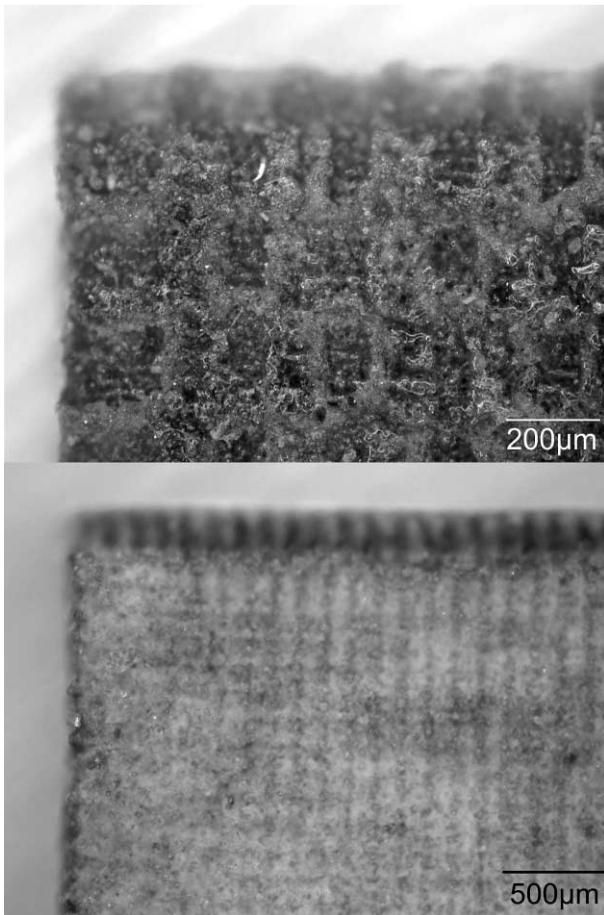


Figure 14. Top: sintered specimen with an amorphous phase of  $\text{SiO}_2$ . Bottom: crystalline phase of a sintered specimen of  $\text{SiO}_2$

**4.2.1. Silicon enriched SiC.** The cube and the miniature gear wheel (figure 16) show high structural resolution and fidelity. The cube has an edge length of 3 mm. The gear wheel is 6mm in overall diameter. The blades have a gauge of 0.5 mm. In table 1 the material composition detected by X-ray diffraction is listed and compared with the composition of the powder. figure 17 is a cross-section view of a comparably sintered body from the same SiSiC material. Both examples are older results. Actually, the ratio of the sintered specimens' material composition for Si/SiC is 1/4.

**4.2.2. Technical SiC.** The first assays showed the inapplicability of the q-switched laser regime for selective sintering of technical pure SiC. As the high pulse peak intensities usually caused disintegration of the material the further experiments were conducted with continuous (cw) laser radiation. With the employment of cw-radiation there is an increased chance of oxidation (Streek et al. 2006),

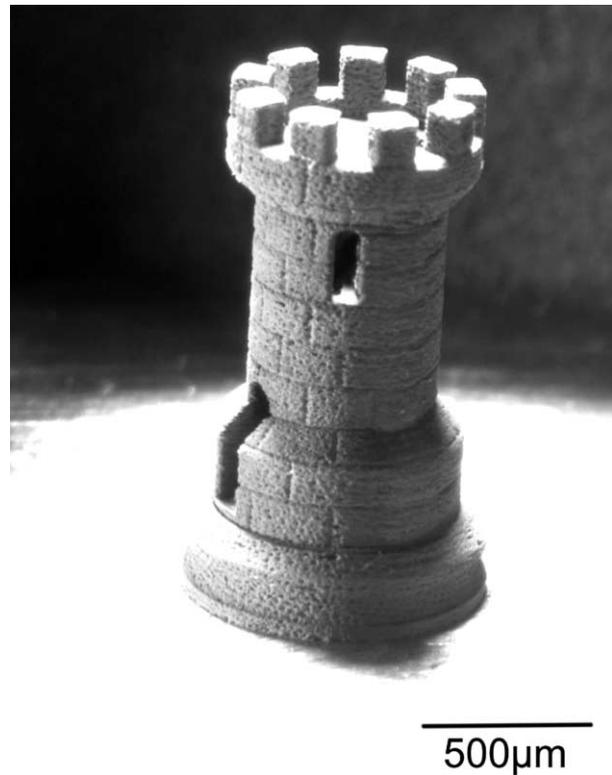


Figure 15. Complex structure consisting of  $\text{SiO}_2$ -ceramic.

therefore the processes were performed under an inert gas atmosphere.

With optimised parameters complex structures are generable. figure 18 presents a part from SiC powder, which, without any sintered support bodies, had been generated with  $63^\circ$  undercuts in good quality. In this case continuous laser radiation (cw) had to be used as the pulse peak intensities usually caused the disintegration of the material. But with the employment of cw-laser radiation there is an increased chance of oxidation, therefore the processes were performed under an inert gas atmosphere.

Another example shown in figure 19 was sintered with a pulsed laser process. A problem in that laser regime is the partly arising laminating. Some layers become detached from each other. For this reason high laser pulse powers must be avoided too.

## 5. Conclusions

A new technology for the selective laser sintering process called LMS has been presented. The LMS -technology was successfully used for the generation of solid bodies made of several metal and ceramic materials. It is applicable for powders with grain sizes from the sub-  $\mu\text{m}$  range up to  $10 \mu\text{m}$ . A variety of differently shaped bodies were

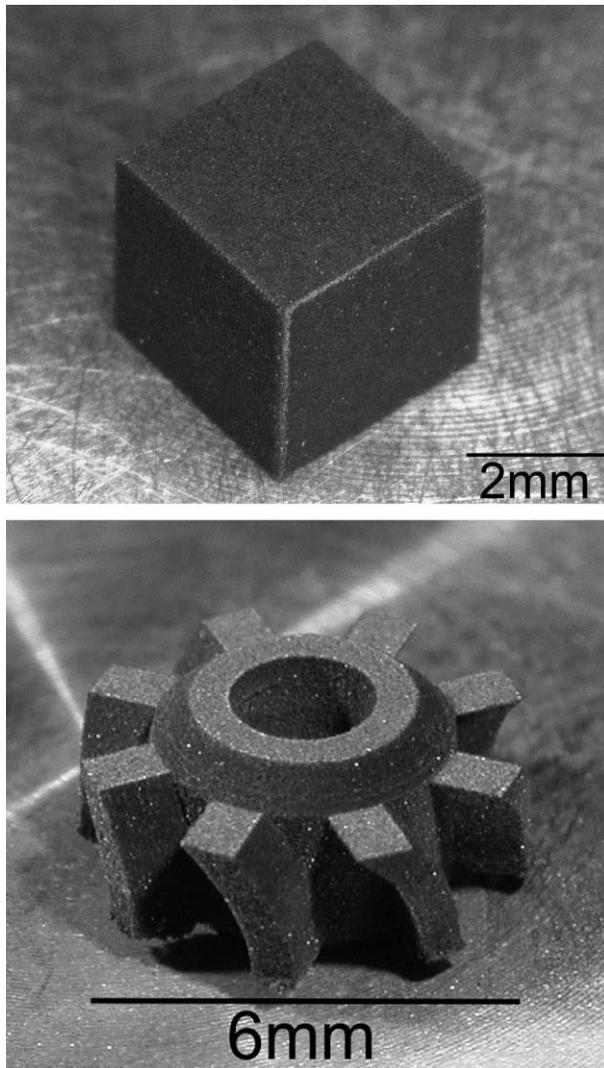


Figure 16. Top: high definition cube consist of SiSiC. Bottom: micro-turbine of SiSiC.

generated with a height of more than 10 mm with a thickness of the sintered layer down to 1  $\mu\text{m}$  and as a consequence with a very good resolution. With this new technology the development of a new class of materials seems possible that is suitable to be processed with laser

Table 1. (a) The components of the applied powder. (b) Portions for the sintered body.

	(a) Powder	(b) Sintered body
Si	41%	63%
SiC	50.8%	37%
3C	0%	5%
15R	?	5%
4H	?	7.6%
6H	?	19.8%
C	8.2%	?

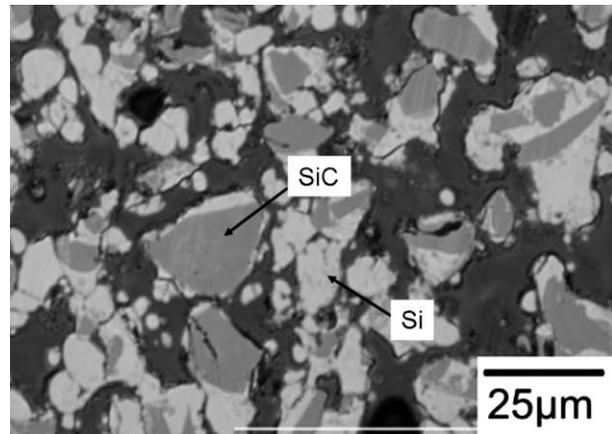


Figure 17. Cross-section view of sintered body from SiSiC powder; green areas: resin matrix.

radiation: new material properties can be expected for gradient and laminated materials.

In the case of metals Al, Ag, Cu, Mo, Ti, W, 80Ni20Cr have been sintered, and various kinds of stainless steel, WCu alloys, and there seems to be no limitation. The resolution is in the range of 15  $\mu\text{m}$ , the roughness has a minimum of  $R_a = 1.5 \mu\text{m}$ . A minimal notch gauge of 10  $\mu\text{m}$  and maximum aspect ratio of 300 was achieved. The relative density of compounds sintered from alloys was higher than 95%.

In the case of ceramic material the best resolution for alumina was in the range of 50  $\mu\text{m}$ . For this material a maximum crushing strength of 1400 MPa and a maximum tensile strength of 120 MPa was measured. There was found nearly no shrinkage ( $< 0.7\%$ ) after heat treatment of the specimens. Special modifications of the sinter regime have to be applied for selective laser sintering of micro bodies made from SiOx and SiC or SiSiC.

#### Acknowledgements

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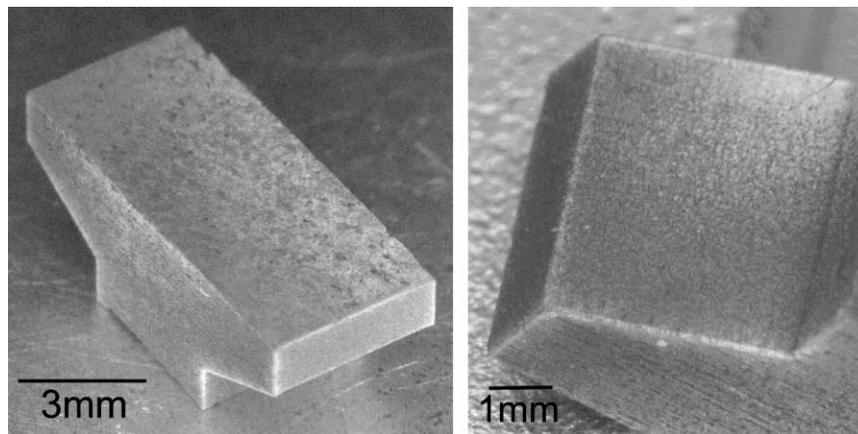


Figure 18. Solid construction unit generated under an inert shield gas with cw-laser action from technically pure SiC powder. Left: overview; right: detail.

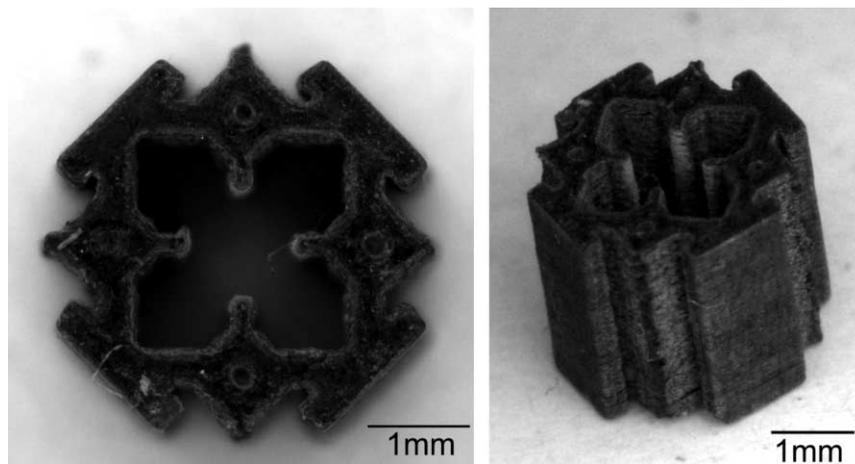


Figure 19. Pulsed laser sintered construction unit from technically pure SiC.

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