

Process Assembly for μm -Scale SLS, Reaction Sintering, and CVD

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Abstract:

A novel device suited for the generation of sintered microparts of metal and ceramics, for reaction sintering and for CVD has been developed and successfully tested. With the production of a functional component it has evidenced professional performance.

The set-up is vacuum tight; unstable substances can be processed under various shield gases and pressures; it is equipped with a device suited to rake thin layers of fine powders as well as slurries.

Sub micrometer powder can be processed in steps of $1\mu\text{m}$ thick sintered layers.

In combination with a proprietary sintering regime, micro parts with a structural resolution of $<30\mu\text{m}$, and aspect ratios of >10 have been achieved.

1. Introduction:

Selective laser sintering (SLS), a familiar technique in rapid prototyping and rapid tooling, was heretofore preferentially applied for the generation of macroscopic freeforms.

Commercial devices with a laser focus diameter of $40\text{-}500\mu\text{m}$ still do not allow generation of microparts smaller than $100\mu\text{m}$. Therefore since its first application, efforts have not ceased to increase the resolving power of SLS, aiming for dimensions in the range of $20\mu\text{m}$. This is beyond the limits of classical chip removing or milling processes.

As SLS is a layer wise material structuring process, the approach of finer details requires thinner layers and consequently powders with smaller grain sizes. The realization of these requirements is not always trivial, as finer grained solids are more reactive than coarse materials. Precautions have to be taken to avoid corrosion of the powder by oxygen or humidity. Moreover, the finer the powder gets, the poorer becomes its "rakeability". The packing of the fine powder layers are very loose as gravitational forces succumb to the inter particle forces. Especially during simple recoating procedures e.g. by sweeping a blade across the modelling platform, the powder forms agglomerates, which are by more than one order of magnitude larger than a single grain. This behaviour can be partly overcome by a special raking strategy; the remaining lack of layer density has to be taken account of by an adequate laser sintering regime.

The Laser Institut Mittelsachsen e.V. in Mittweida, Germany, has developed a procedure^{1,2/} and a sintering machine, which makes feasible the generation of solid and structured parts out of metals and ceramics by direct selective laser sintering. To overcome the difficulties from oxidation and humidity, the complete process was transferred into a vacuum tight chamber^{3/}. The obtained structures show a resolution of less than $30\mu\text{m}$ at aspect ratios >10 , and a minimal roughness of $3.5\mu\text{m}$ can be achieved.

2. Process Assembly and Performance

2.1 Process Assembly:

The process assembly [Fig.1] consists of the sintering chamber (SC) [Figs.2], an attached turbo molecular vacuum pump, a ScanLab beam scanner with a scan field of $25\text{x}25\text{mm}$, a Q-switched Nd:YAG – laser ($\lambda = 1064\text{nm}$) with an output of $0.1\text{-}10\text{W}$ in TEM_{00} mode and $0.5\text{-}50\text{kHz}$ pulse frequencies, gate valves for various shielding and reaction gases as well as the power supply and the control unit for the coating and positioning bench (CPB).

The coating and positioning bench (CPB) - the core of the SC, where the sintering takes place - is mounted inside a vacuum tight stainless steel casket, the lid of which has an integrated

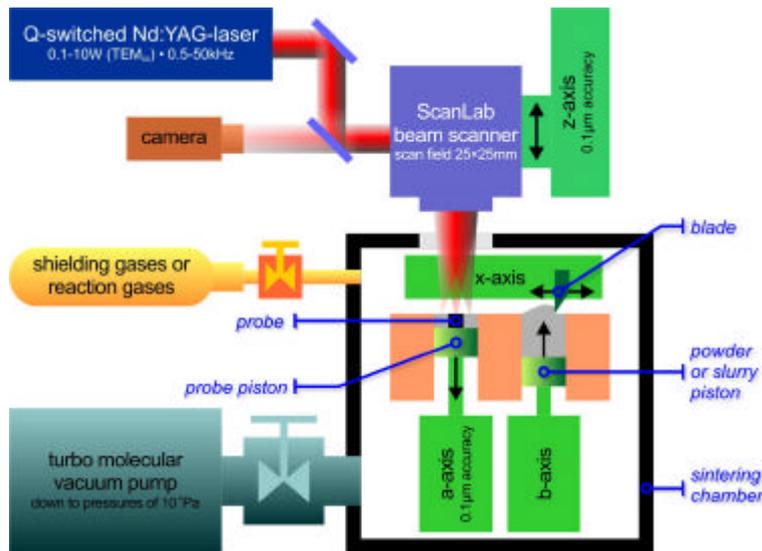


Figure 1: Schematic set-up for laser microsintering

platform. The platform is positioned horizontally and has two vertical cylindrical bores for the powder piston and the probe piston. Each of it has its separate drive [Fig3].

quartz glass window with transmission for the applied laser radiation. The casket has electrical feed throughs for the sintering platform and an internal process observation camera. Several valves allow for the exchange of the shielding and reaction gases; at a major and a minor connection respectively, the pump and a manometer are attached to the SC.

The CPB has an aluminium frame, holding three piezo ceramic drives with a resolution of 0.1µm, and the sintering

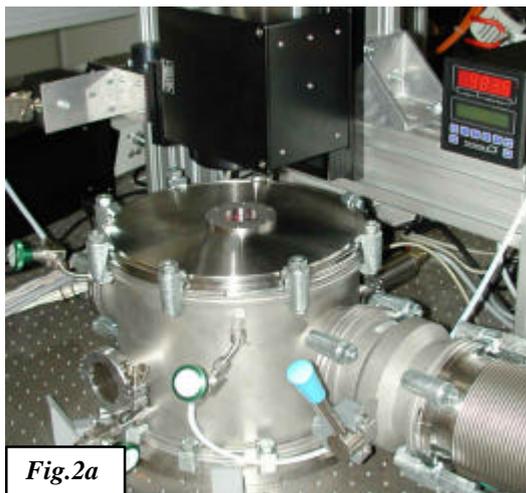


Fig.2a

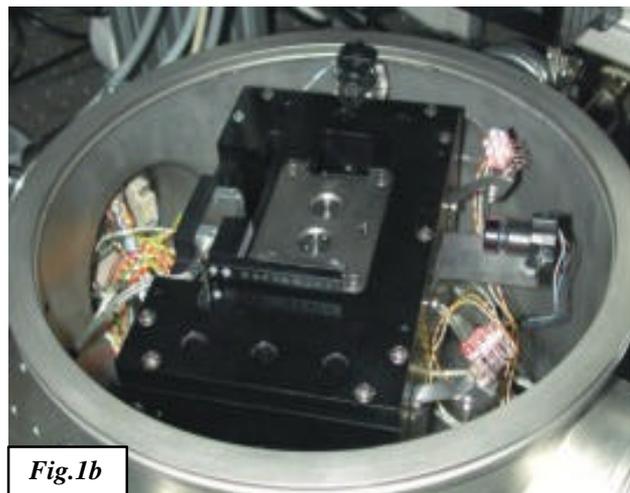


Fig.1b

Figures 2: View of the SC during operation (a) and after removal of the lid (b).

With the third drive a proprietary powder rake, which has the shape of a ring with a sharpened edge, is swept across the platform. Because of its shape the rake also serves as an intermediate powder reservoir [Fig.4a]. The position of the blade is manually adjustable, it is supposed to run as low over the platform surface as possible. Thus the waste of powder during a generation cycle is minimized. The pistons are tight for powders and liquids, which allows to process also emulsions and ceramic slurries [Fig. 4b].

The SC can be evacuated by the attached turbo molecular pump down to pressures of 10^{-3} Pa and it can be charged with shielding gases or reaction gases at any pressure in the range between 10^{-3} Pa up to 4×10^5 Pa. A second – chemically resistant – pump

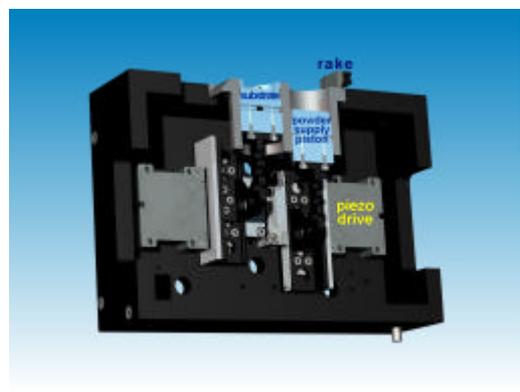


Figure 3: Cross sectional view of the CBS

can be connected to the chamber and, with a system of flow controls and pressure reducers, reaction gases can be flushed through at pressures of $\approx 1\text{Pa}$, which makes the SC applicable for laser chemical vapour deposition (Laser CVD).



Figures 4: The top of the CBS with the ring shaped rake.
a: Substrate lowered and uncoated, rake filled with metal powder. b: Substrate coated with metal powder. c: Substrate coated with slurry, rake loaded with

The proprietary software *IVS STL Converter (Version 1.0)* that was developed by *IVS SOLUTIONS AG*, and *3D Micromac AG* especially for this purpose controls the sinter process. STL – data can be processed with a high resolution on a micrometer scale. Especially curves are executed at fast rates with high precision. Outline and filling parameters can be adjusted arbitrarily. Another - self-developed - program allows flexible control of the raking routine. The programs are accessed by the software via an interface, which facilitates automatic performance of a complex SLS - process. Continuously repeated calibration of the scanner is integrated into the software accounting for the fidelity and precision of the technique, even at high aspect ratios.

2.2 Materials:

For the generation of metallic free forms single component powders were used (Table 1), in addition, metal sintering was performed with mixtures of copper and tungsten.

Table 1: Processed Metal Powders and their Grain Sizes

Metal	Tungsten	Aluminium	Copper	Silver
Grain Size	300nm	3 μm	10 μm	2 μm

All metals are relatively inert materials at low and normal temperatures. When processed with laser radiation under a normal atmosphere, however, most of them show considerable oxidation.

Presently direct sintering of ceramics is probed with aluminium nitride powder and a porcelain raw material as a nonoxide ceramic and an oxide ceramic with a glassy component respectively.

Selective reaction sintering is being done with aluminium powder under nitrogen.

The results presented in this article confine to selective sintering of metal powders, especially tungsten.

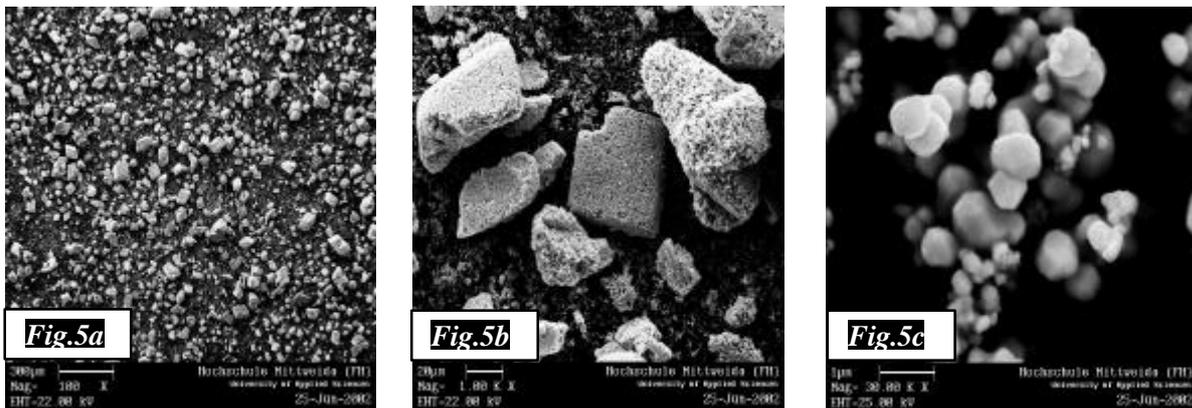
2.3 Process:

The process atmosphere:

To provide the proper atmosphere for the process, the SC is evacuated to 10^{-3} Pa. Depending on the condition of the powder the vacuum is applied for several hours to allow desorption of water. Subsequently, the chamber is charged with the shielding gas at the appropriate pressure between 10^4 and 10^5 Pa. Usually the gas does not need flushing or exchange in the course of a process even if this extends over more than one day.

The raking procedure:

As mentioned above, the raking of a thin layer of fine grained powder causes problems, because the material does not sediment in a dense packing but – partly supported by the raking – forms agglomerates which in the case of a sub micrometer tungsten powder often occur in the shape of polyhedrons with a preference for certain angles. The agglomerates, which are approximately an order of magnitude larger than the grain size, do not pack densely either. The mass of the particles is too low for gravity to suffice for a dense sedimentation. Figures 5 show the agglomerated consistency of tungsten powder with an average grain size of $0.3\mu\text{m}$ and some of the few unagglomerated grains.



Figures 5: SEM views of tungsten powder with an average grain size of $0.3\mu\text{m}$ at different magnifications.

To overcome this drawback a special raking regime was developed to generate a thin layer by first applying a thicker one which is sheared off by successive raking from opposite directions. The nature of the interparticular forces is not quite clear, but obviously the amount of absorbed water plays a certain role, as exposition of the powder to a vacuum of 10^{-3} Pa for several hours improves the result of the raking procedure. The raking speed was $50\text{mm}\cdot\text{s}^{-1}$. Still, however, the density of the resulting layer is very poor, estimations are in the range of 15%, so that further condensation has to be achieved during sintering.

3. Results:

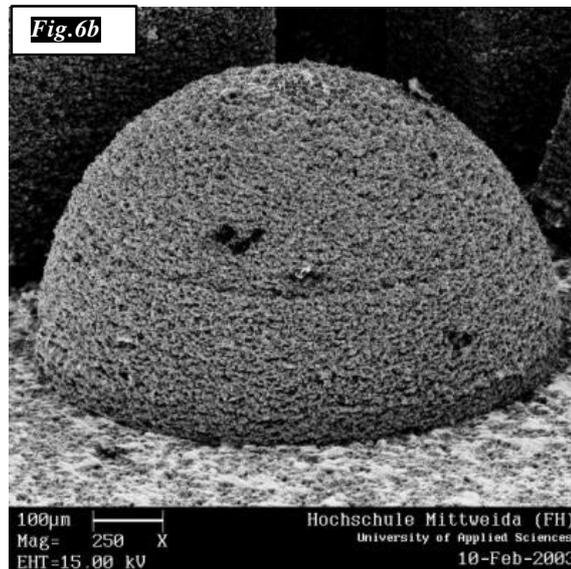
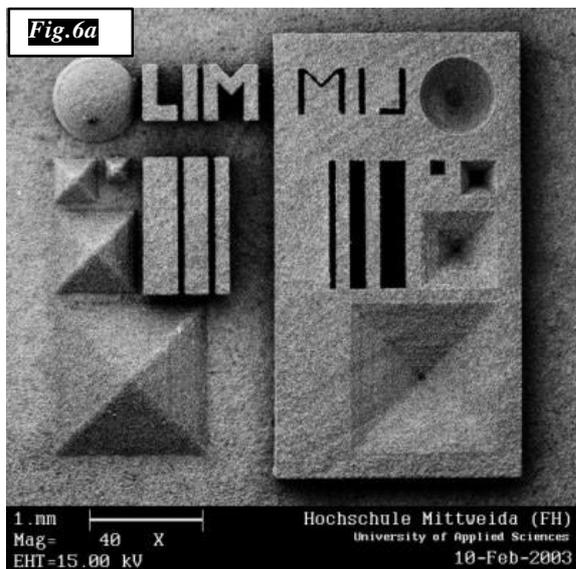
With a special sintering regime prismatic and tapered microstructures were generated from tungsten and other metal powders [Figs.6].

After the method had proved reliable to generate micro freeforms with a sufficient fidelity, a tool component [Fig.7] was built to fulfil a function in an industrial routine. The part (made of tungsten) is 10mm at its longest dimension. It is partly solid; a slit with an open width of $480\mu\text{m}$ and a length of 3.75mm is connected to a circular window (diameter: 1mm) by a tunnel through the solid body.

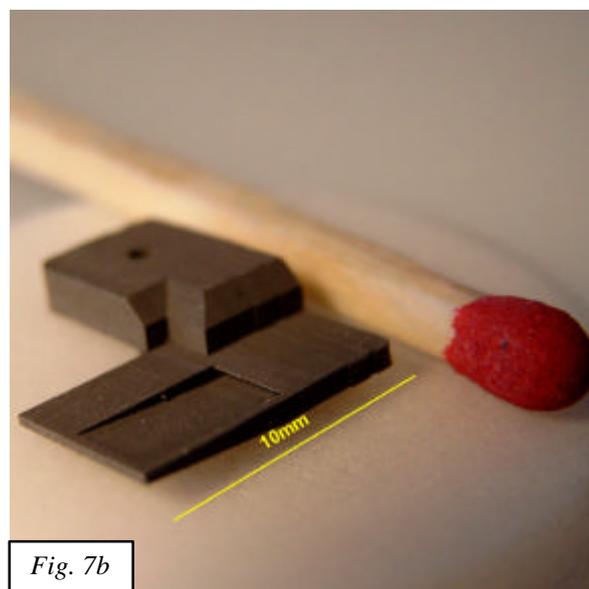
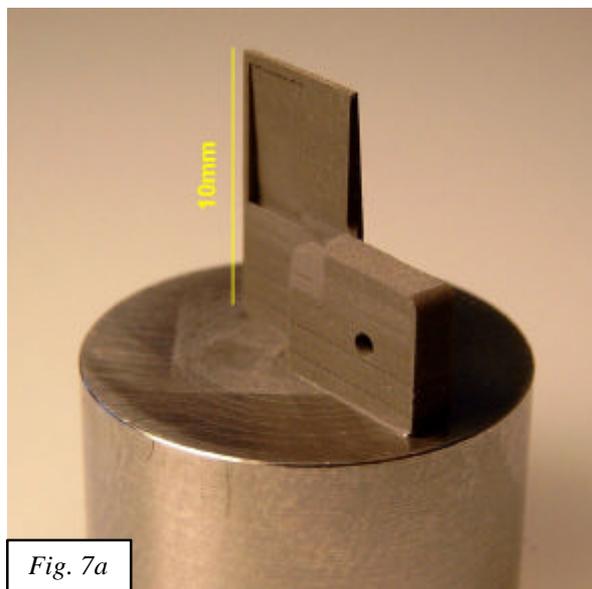
Our client, by whose courtesy we are able to present the views of the sinter product, reported the roughness values shown in Table 2.

Table 2: Surface Roughness (R_a)

Surface Type	horizontal	vertical	separation cross section
Grain Size	$5\mu\text{m}$	$3.5\mu\text{m}$	$7\mu\text{m}$



Figs. 6: SEM views of prismatic and tapered micro structures from $0.3\mu\text{m}$ tungsten powder.



Figures 7: A functional freeform was generated, loosely attached to a stainless steel substrate (a). The freeform was disassembled easily from the substrate.

Conclusion and Perspectives:

A novel set up facilitates the generation of micro freeforms by direct selective laser sintering, as it provides for the handling of the corresponding fine-grained powders. A hermetically closed sinter chamber and a special rake take care of the enhanced reactivity and the extraordinary coating behaviour of the materials. Micro-freeforms from a number of metals have already been obtained with the set-up applying a special laser sintering regime. With a slightly different approach, the technique is also applied for the selective sintering of ceramics and composite materials. The ideas and applications of the innovation are registered in Germany as patents and utility models.

Appreciations:

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Patent Pending, reference number 199 52 998.1, date of application 01.11.1999.