Selective Laser Micro Sintering with a Novel Process

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Abstract:
Microparts with a structural resolution of <30µm and aspect ratios of >12 have been generated by selective laser sintering. The technique includes sintering under conditions of vacuum or reduced shield gas pressures. In a novel set-up the material is processed by a Q-switched 1064nm Nd-YAG laser after a special raking procedure. The procedure allows the work pieces to be generated from powders of high melting metals like tungsten as well as lower melting metals like aluminium and copper.
Contingent on the parameters, the generated bodies are either firmly attached to the substrate or can be dissevered by a non-destructive method.

1. Introduction:

Selective laser sintering (SLS), a familiar technique in rapid prototyping and rapid tooling, has heretofore been preferentially applied for the generation of macroscopic freeforms. Commercial devices with a laser focus diameter of 40-500µm still do not allow generation of microparts smaller than 100µm. Therefore since its first application, efforts have not ceased to increase the resolving power of SLS, aiming for dimensions in the range of 20µm. This is beyond the bounds of classic chip removing or milling processes.
Compared to still higher resolving techniques, SLS still bears the advantages of relatively low production costs and short processing times for uniques or small sample numbers. Sintered microstructures with prismatic or tapered shapes can be applied as electrodes for electro erosion, as tools for direct shaping of plastic materials or as molds for injection molding. Furthermore freeform - meaning “tool-independent” - undercuts and hollows can be realized easily, allowing e.g. the fabrication of miniature tools and components with hydrodynamic functions. Therefore SLS remains an attractive tool for the mentioned size range.
As SLS is a layer wise material structuring process, the approach towards 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 “rakeablity”. 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 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 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 /1/. The obtained structures show a resolution of less than 30 µm and a minimal roughness of 3.5µm can be achieved.

2. Process Performance

2.1 Process Assembly:

The sinter process takes place in a sinter chamber /2,3/ that can be evacuated by an attached turbo molecular pump down to pressures of 10^{-3}Pa and can be charged with shielding gases or reaction gases at any pressure in the range between and 10^{-3}Pa up to $4x10^{5}$Pa. It provides a quartz glass window for the incident beam and is suited for the proper handling of materials with enhanced reactivity and extraordinary coating behaviours. Via a system of flow controls and pressure reducers, reaction gases can be charged or flushed through at pressures of =1Pa, which makes the sinter chamber applicable for laser chemical vapour deposition (Laser CVD).

The source of radiation is a Q-switched Nd:YAG – laser (? = 1064nm) with an output of 0.1-10W in TEM_{00} mode and 0.5-50kHz pulse frequencies; the beam is guided into the chamber and along the sinter track on the powder layer by a ScanLab scanner.

The presented results were achieved with the above-described laser in the pulse regime. Micro freeforms generated with continuous wave radiation and different wavelengths will be the subject of upcoming presentations.

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	3µm	10µm	2µm

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 as a nonoxide ceramic and a porcelain raw material as an oxide ceramic with a glassy component respectively.

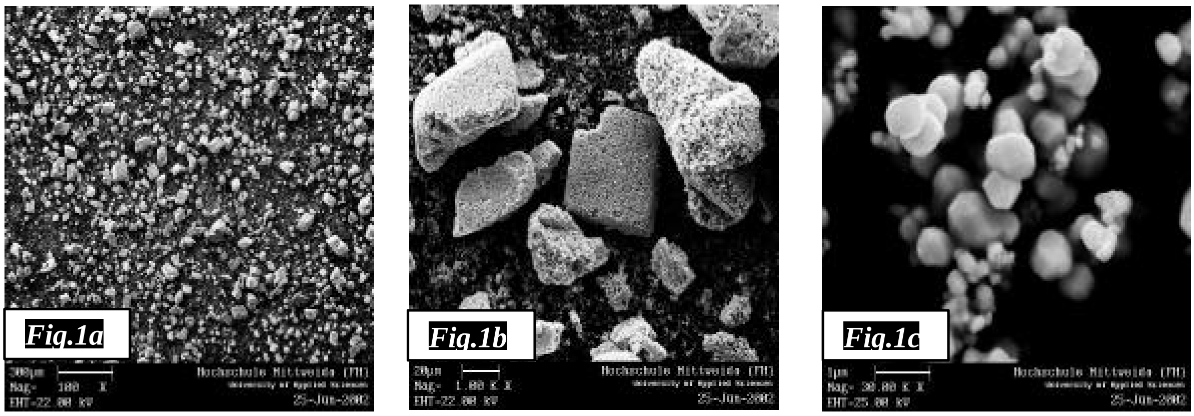
Selective reaction sintering is being done with aluminium powder under nitrogen.

2.3 SLS Process:

The process atmosphere:

To provide the proper atmosphere for the process, the sinter chamber 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 [*Figs.1*] 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. 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 $50 mms^{-1}$.



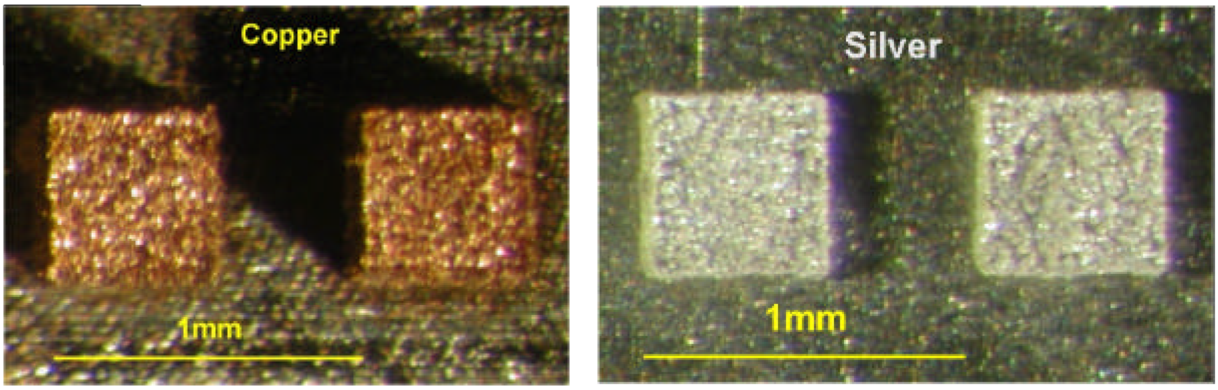
***Figures 1**: SEM views of tungsten powder with an average grain size of 0.3µm at different magnifications.*

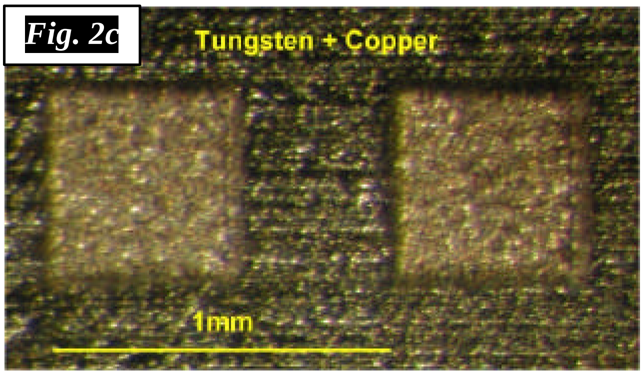
Still, however, the density of the resulting layer is very poor, estimations are in the range of 15%, so that further condensation has to be achieved during sintering.

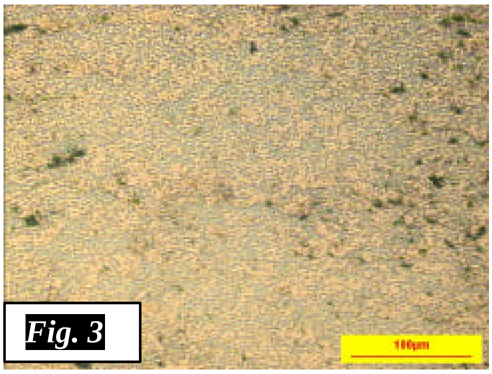
Sintering:
Laser pulses with powers from 0.5kW-2kW were applied at repetition rates in the range of 5-20kHz. The cross sections of the microparts are processed with the pulsed radiation in a way that the pulses are distributed evenly across the selected areal segments. The resulting solid area is not a closed coating of metal, but is more a network of craters or wedges that root about 10µm below the mean surface level with crests above between 1 and 3µm. This effect accounts for the higher quality of the generated vertical surfaces compared to the horizontal surfaces. Specific regimes are applied for bottom and top horizontal surfaces respectively. Metallic micro free forms can be either fused to the metal substrate or attached to the substrate surface by narrow sinter necks, frail enough to be sheared off without destruction of the generated free form but stable enough to fix the part throughout the raking and sintering process. Also, for stable positioning, the adjacent powder zone is processed with low power embedding the freeforms in a crust that can be removed completely by subsequent ultrasonification.

3. Results:

Figures 2 show cuboids from copper and silver powders as well as from a tungsten/copper powder mixture. Highest densities of 90% and above have been achieved from copper/tungsten powder mixtures with a mass ratio copper/tungsten of 75:25 [*Fig.3*]







Figures 2: *Cuboids sintered from metal powder: copper* ***(a)****, silver* ***(b)*** *and copper/tungsten* ***(c)****.*

Figure 3: Cross sectional view of a sintered mixture of 25wt% tungsten and 75wt% Copper that takes the role of the liquid phase during sintering. The density of the sintered body exceeds 90%.

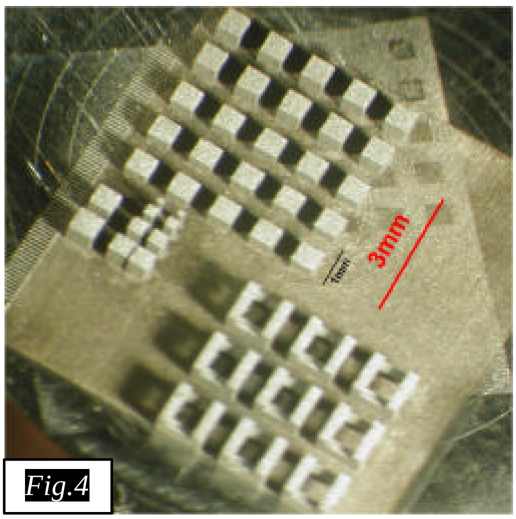
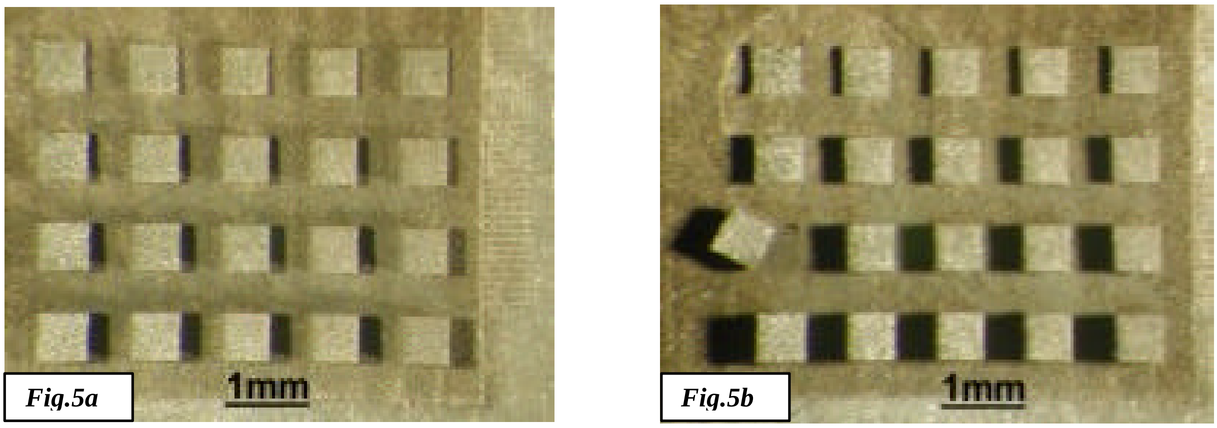


Figure 4: *Array of tungsten cuboids and bridges, firmly attached to the substrate.*

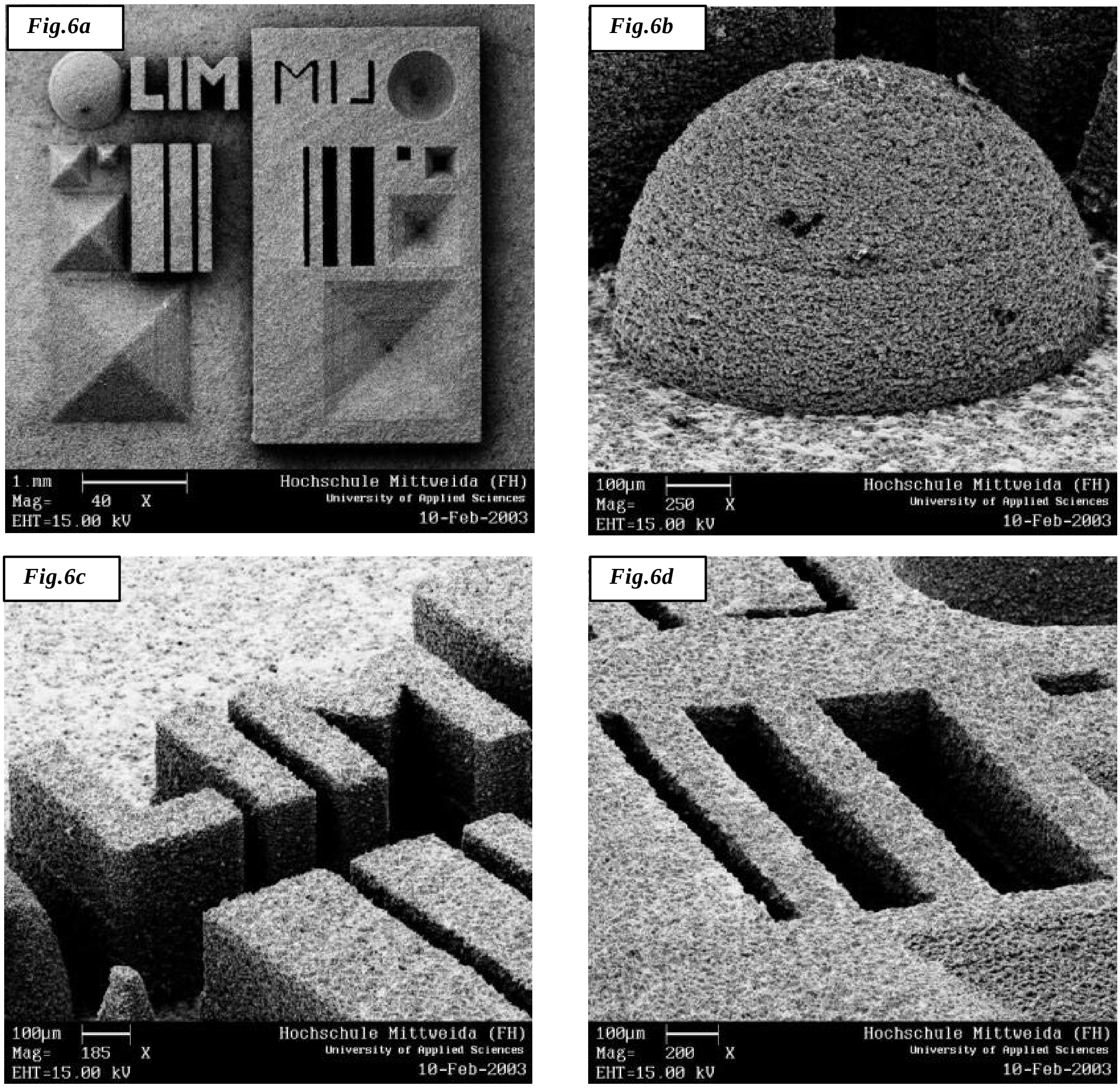
Cuboids and undercut microstructures were generated from tungsten metal powder.

Figures 4 and 5 show an arrangement of cuboids and bridges fixed to the substrate [*Fig.4*] and another set of cuboids separably attached to the stainless steel base plate [*Fig5a, b*]. Figure 5b shows a microbody that was split off the substrate by a slight chop with a spatule.



Figures 5: *Array of tungsten cuboids, separably attached to the substrate; after removal of the powder bed* ***(a)*** *and subsequently after loosening a single cuboid with a spatule* ***(b)****.*

Figure 6a shows SEM views of a tungsten structure with a depth of 400µm. It contains notches with a width of <40µm, a resolution that cannot be achieved by micro-milling techniques.



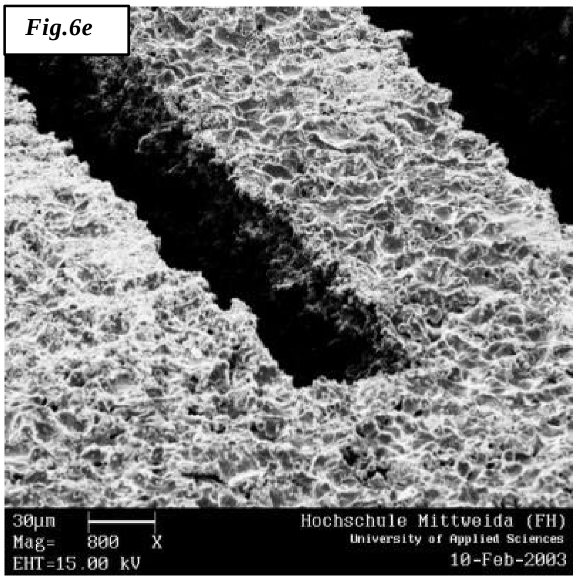


Fig.6e

Figures 6:
SEM views of a multishape test structure sintered of tungsten powder; the diagonal hatch of the surface texture (a) as well as local scourings of the upper surface (d) are due to abrasion by the rake after occasional slight failures of the probe piston. The narrowest achieved notches have a width of 40µm (c,d,e).
The structure is 400µm high.

As mentioned above, the roughness of the horizontal surfaces is higher than the roughness of the vertical surfaces. The two halves of the structure were not intended to be complementary. Figures 6b-e show details.

After the method had proved reliable to generate micro freeforms with a sufficient fidelity, a tool component was built to fulfil a function in an industrial routine. The part (also made of tungsten) is 10mm at its longest dimension. It is partly solid; a slit with an open width of 480µm and a length of 3.75mm is connected to a circular window (diameter: 1mm) by a tunnel through the solid body. The roughness values shown in Table 2 were reported by our client, by whose courtesy we are able to present views of the sinter part in Figures 7a,b.

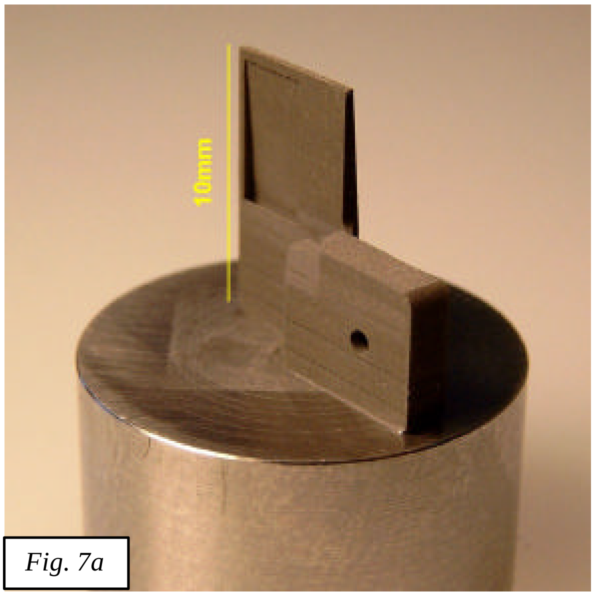


Fig. 7a

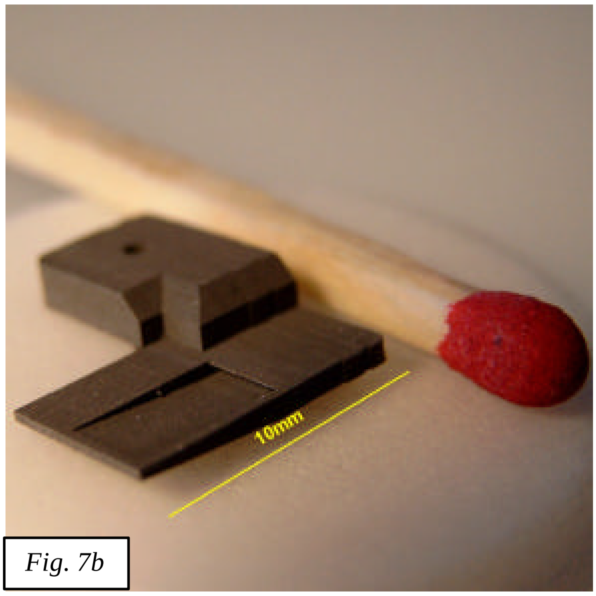


Fig. 7b

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

Table 2: Surface Roughness (R_a)

Surface Type	horizontal	vertical	separation cross section
Grain Size	5µm	3.5µm	7µm

For the fundament of the freeform, parameters were chosen to allow easy detachment and as we were informed, holding the part between thumb and forefinger, it could be separated from the substrate easily by a gentle twist.

Conclusion and Perspectives:
A novel set up and processing regime, have extended the applicability of freeform generation by selective laser sintering into a range of structural resolution and reproduction fidelity heretofore unachieved /4,5,6/. The results presented here are obtained from powders of tungsten copper, silver and tungsten/copper mixtures. They prove the technique an effective and versatile method for rapid micro tooling.
With a slightly different approach, the technique is also applied for the selective sintering of ceramics and composite materials; this will be subject of forthcoming publications.
The ideas and applications of the innovation are registered in Germany as patents and utility models.

Appreciations:
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