Selective Laser Sintering of Zirconium Silicate

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Abstract

The Fraunhofer Institute of Production Technology (IPT) and the Fraunhofer Institute of Laser Technology (ILT) have joined forces in a project dedicated to Selective Laser Sintering (SLS) of metals and ceramics, funded by the German Government and 6 industrial partners.

Selective Laser Sintering of zirconium silicate as a ceramic material used for investment casting shells and cores is an attractive alternative to the conventional, time-consuming way of producing these shells from a wax master. This paper will present current process results concerning laser sintering of shells made from zirconium silicate and explain the related potentials and benefits.

Acknowledgements

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1 Investment Casting

Investment casting is an industrially well established casting process for the production of complex metal parts for small- to large-sized batches. A pattern, usually made from wax by injection molding, is used to create a coating by dipping the pattern in a ceramic slurry and drying the coating. This procedure is repeated up to nine times to create a shell, consisting of ten layers with an overall thickness of about 6 mm. The ceramic shell is then fired in an oven to burn out the pattern and sinter the material to gain additional strength. A metal is poured into the shell to cast the metal positive; the process ends with removal of the shell as well as the runner systems and additional cleaning. Both the pattern and the shell are destroyed during the casting procedure (lost pattern, lost form casting process). An advantage of investment casting is that practically all metals may be processed (König, 1990).

Investment casting is often used in conjunction with Rapid Prototyping (RP). Usually, RP is employed to create the master pattern; QuickCast from 3D Systems and Selective Laser Sintering of polystyrene or polycarbonate are good examples (Jacobs, 1995, Venus, 1995).

However, time is only saved during production of the pattern, yet the casting process itself takes about two weeks due to the necessary step of drying the ceramic slurry coatings that are repeatedly being applied. Thus, the direct production not of patterns but of ceramic shells by RP yields great benefits since creation of both a pattern and a shell is superfluous. The Direct Shell Production Casting process developed at MIT and in application at Soligen already follows this route utilizing 3D Printing (Cima, 1991).

2 Experimental Results

Selective Laser Sintering is a process also capable of generating parts from ceramic powder (Esser, 1996, Langer, 1996, Lakshminarayan, 1992, Griffin, 1995, Lee, 1995, Vail, 1993). The materials most often used in investment casting are aluminum silicates, zirconium silicates, alumina and silica. From initial tests, zirconium silicate was found suitable for use with Selective Laser Sintering.

Figure 1 shows first experimental results that were obtained by Selective Laser Sintering of a zirconium silicate (-125 mesh, particle size < 119 µm, irregular particle shape) using a cubic test geometry (10 x 10 x 10 mm^3) and varying laser power from 14 to 88 W, scan speed from 100 to 800 mm/s and hatch spacing from 0.05 to 0.4 mm. Each layer was exposed to the laser light once and scanning was performed in just one direction. The slice thickness was set to 0.2 mm, the machine used was an EOSINT M 160 with a CO_2 laser source. The substrate used was a granite plate which was found more suitable for this purpose than other materials, e.g. metals, due to the low thermal conductivity and the good adhesion characteristics.

- processing is possible with an energy density of E=2 to 6 J/mm^2
- agglomerated particles formed from molten material cause break-outs while wiping
- little curling

material
zirconium silicate
grain -125, particles < 119 µm
machine parameters
laser power P_L = 14 to 88 W CO_2
scan speed v_s= 100 to 800 mm/s
hatch spacing h_s=0.05 to 0.4 mm
exposure x-direction
thickness of layer 0.2 mm
test part geometry
10 x 10 x 10 mm^3

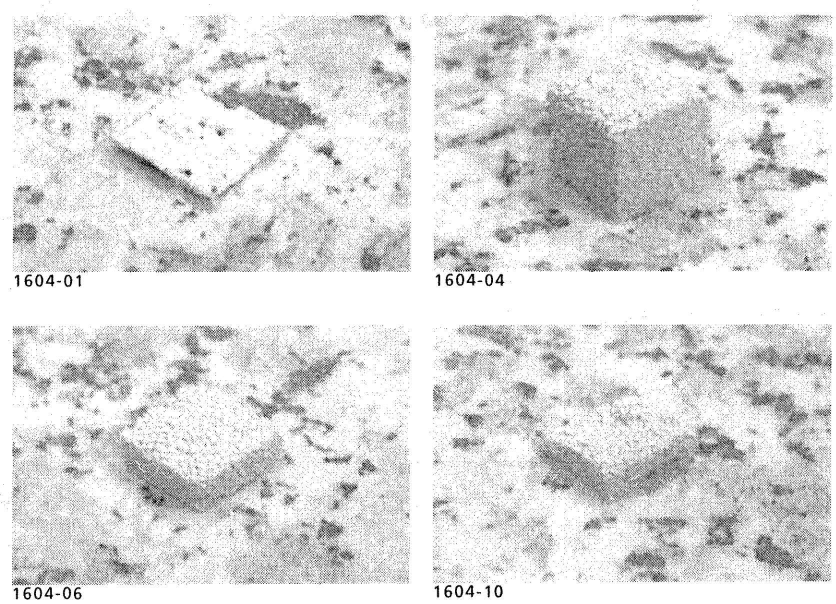


Figure 1 SLS of zirconium silicate – experimental results

The experiments were successful for some parameter combinations (see figure 1, upper right hand corner: laser power P_L=88 W, scan speed v_s=100 mm/s, hatch spacing h_s=0.4 mm) while others either led to irregular, totally molten shapes (see figure 1, upper left hand corner: laser power P_L=88 W, scan speed v_s=100 mm/s, hatch spacing h_s=0.05 mm) or to agglomerations that were destroyed when a new layer of powder was applied by the wiper (see figure 1, lower left hand corner: laser power P_L=88 W, scan speed v_s=200 mm/s, hatch spacing h_s=0.1 mm; see also figure 1, lower right hand corner: laser power P_L=88 W, scan speed v_s=400 mm/s, hatch spacing h_s=0.1 mm).

After these initial tests to roughly find suitable parameter combinations for the successful SLS of the material, further experiments were conducted with a powder mix of DIN 70 (particle size < 90 μm) and -125 mesh (particle size < 119 μm), this time using a larger test geometry (20 x 20 x 8 mm^3) and scanning one layer in x- and y-direction. The purpose for using a powder mix was to increase the portion of smaller particles in the powder providing an increased amount of liquid phase during sintering, since smaller particles need less energy to melt than larger particles. A further decrease in grain size of the powder was not possible due to an increased amount of agglomerations, making an even spread of new powder layers with the wiper impossible.

Some basic mechanisms were found to prevail during SLS of zirconium silicate as illustrated in figure 2. An increase in laser power leads to an increase in the density of the specimen, since more energy is delivered into the powder and larger melt pools fill up the porous structures of previous layers (see figure 2, upper left hand corner). However, surface roughness also increases since the molten particles tend to form larger spherical structures due to their effort to reduce the free enthalpy by optimizing the ratio between the area of free surfaces and the related volume. Figure 3 is an illustration of this effect; a high speed camera was used to monitor SLS of zirconium silicate, this time on a test facility equipped with a Nd:YAG laser. Laser power was varied from 97 W to 120 W to 161 W, making the effect of formation of increasingly larger structures with increasing laser power visible.

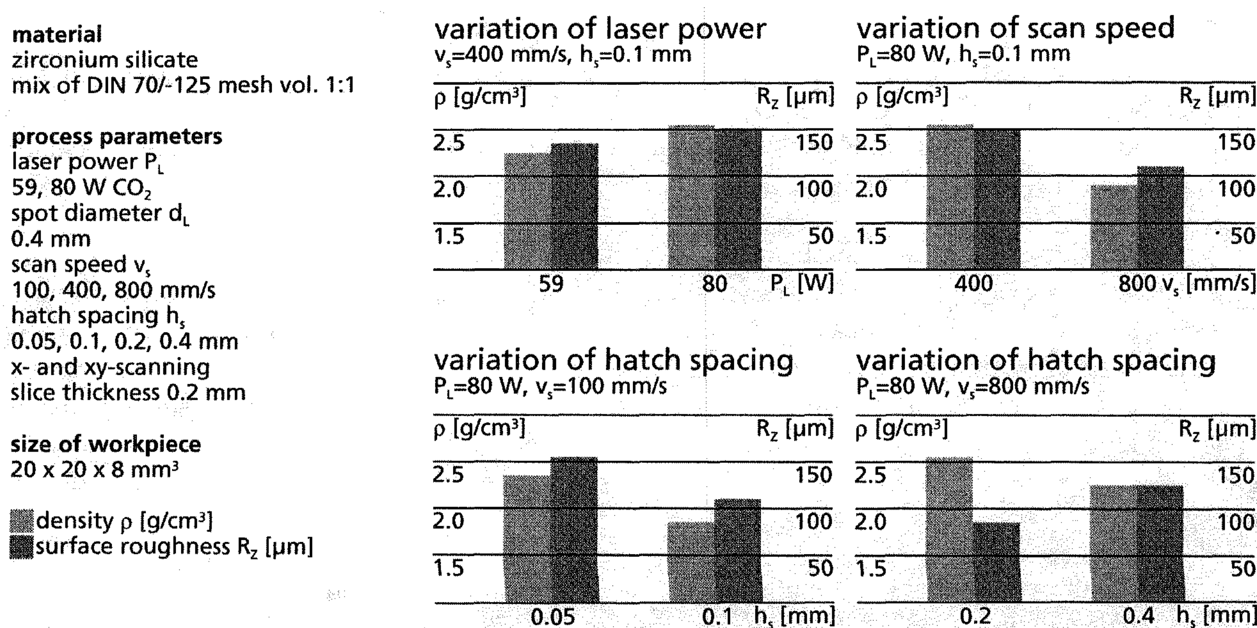


Figure 2 Basic mechanisms of SLS of zirconium silicate

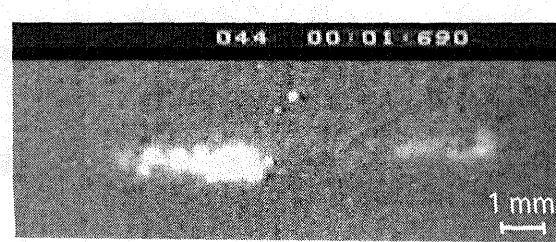
material
zirconium silicate
mix of DIN 70/-125 mesh
vol. 1:1

process parameters
laser power P_L varying
from 97 to 161 W
Nd:YAG laser
scan speed
v_s=100 mm/s
hatch spacing
h_s=0.4 mm
exposure of a single
layer in x-direction
layer thickness is 2 mm
on aluminum plate

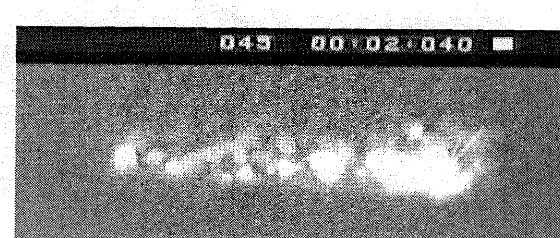
size of workpiece
10 x 10 mm^2

camera parameters
400 shots/s
time notation
mm:ss:1/1000sss

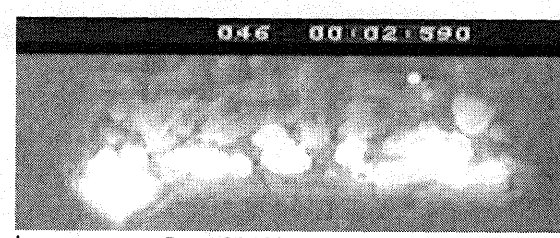
analysis
the influence of laser
power on melting
characteristics of the
powder is evident



laser power P_L=97 W



laser power P_L=120 W



laser power P_L=161 W

Figure 3 SLS of zirconium silicate – high speed video pictures

Increasing scan speed has the opposite effect; because less energy is delivered into the powder bed and the velocity of the laser beam on the part surface is higher, less material is melted and less time is available for the formation of agglomerations, see figure 2. Thus, density decreases and surface finish improves.

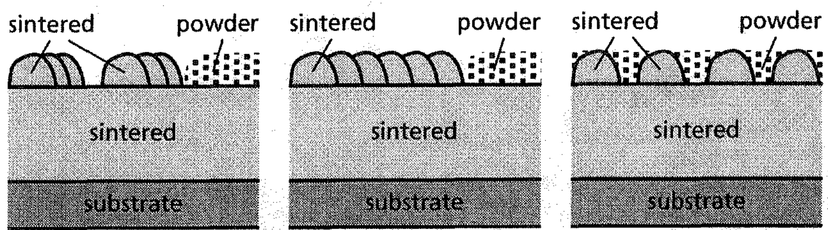
A variation of hatch spacing leads to two different effects, see figure 2 and figure 4. For the case when hatch spacings are small compared to the laser diameter ($h_s << d_L$), the molten powder particles form separate larger beads of high density leading to an overall high density of the part, yet the surface roughness also increases. An optimum combination of density and surface roughness is obtained for hatch spacings that are comparable to the size of the spot. For hatch spacings larger than the diameter of the laser, the molten beads of material are not connected to each other, but the gap is filled with unsintered powder, resulting in a rough surface and low density.

Of all the parameter combinations that led to a stable sintering process, the maximum density achieved was 55.3% of theoretical density (P_L=80 W, v_s=400 mm/s, h_s=0.1 mm, xy-exposure). However, density is not an important quality criterion for investment casting shells. Actually, a certain porosity is desirable to allow gases to diffuse out of the shell when the hot metal is cast. Rather, process stability and repeatability becomes an important criterion. Parts sintered from zirconium silicate are extremely brittle and a collision with the wiper blade during the process due to agglomerates will almost always lead to a destruction of the workpiece.

bead forming, balling effect

- the molten powder particles tend to form larger spherical structures to reduce their free enthalpy
- the wettability characteristics depend on (among other things) the material used and the process temperature
- small grain sizes usually lead to a reduction of this effect, however the powder material may not be processable, i.e. wipable

$h_s << d_L$	$h_s \approx d_L$	$h_s > d_L$



$h_s << d_L$	$h_s \approx d_L$	$h_s > d_L$
the powder particles form separate larger beads of high density with large gaps in between	the powder particles form closely connected, small beads of equal size	the powder particles form unjoined, small beads with powder filling the gaps in between

Figure 4 Basic mechanisms of SLS of zirconium silicate – effect of hatch spacing

3 Practical Testing

A stable parameter combination was found to be a laser power of 80 W, a scan speed of 400 mm/s, a hatch spacing of 0.1 mm using xy-exposure. Vat-like parts were built with these parameters for initial investment casting tests, see figure 5. To improve surface finish and test the effect of infiltration and pre-burning, parts were either left untreated, fired or coated with a $ZrSiO_4$ or $AlSiO_2$ slurry (particle size -325 mesh) and fired. In all cases, the material could successfully be cast into the shells. The infiltration led to an improvement of surface finish, however the small particles in the infiltration slurry do not penetrate the part but clog up the small pores, leading to a sieve-like effect, covering the surface of the workpiece and leading to undesirable dimensional inaccuracies. Firing the specimen before casting the metal does not lead to a measurable advantage in surface finish or density.

Finally, test parts (tensile test bars, articulated lever) were constructed and runner systems were added. The parts were triangulated and shells were created by offsetting the outer surfaces (6 mm) of the parts using STL file manipulation software. Several shells were built (build time: about 20 h each) on the EOSINT M 160 employing the parameters mentioned above. The internal structures were reproduced without affecting process stability. The shells were cleaned externally and internally from excess powder with compressed air and water and then preheated for 3 hours to 380° C. The metal cast was an AlSi 7 with a melt temperature of 750° C. The shells were fully filled and all features were reproduced, see figure 6 and figure 7. At this stage of development, the primary goal was to prove the suitability of the SLS process for the production of shells made from zirconium silicate, thus attainable accuracies were not measured. The surface of the cast part was an accurate reproduction of the surface of the ceramic negative, thus surface roughness was in the range of 100 to 150 μm.

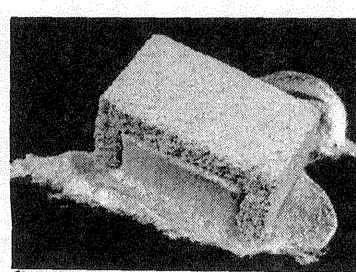
material
zirconium silicate
mix of DIN 70/-125 mesh vol. 1:1

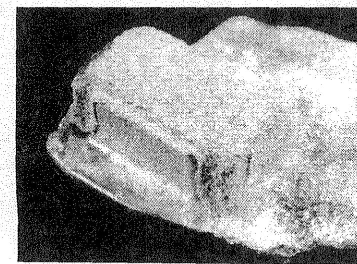
process parameters
layer thickness 0.2 mm
exposure in xy-direction
CO_2 laser
spot diameter d_L=0.4 mm

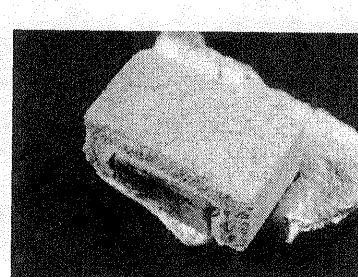
laser power P_L=80 W
scan speed v_s=400 mm/s
hatch spacing h_s=0.1 mm
energy density E=4 J/mm^2
line energy E_L=0.5 J/mm^2

test piece geometry
30 x 20 x 14 mm^3
wall thickness 6 mm

temperature
burning temperature 1000°C
preheating temperature 380°C
melt temperature 750°C


fired


coated with $ZrSiO_4$, fired


coated with $AlSiO_2$, fired

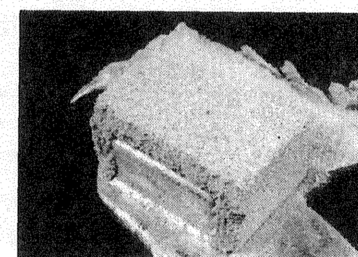
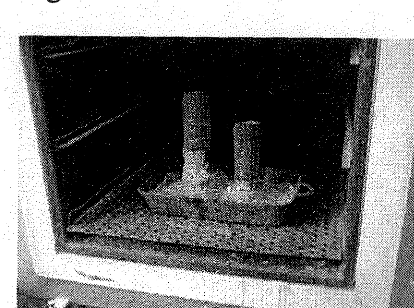
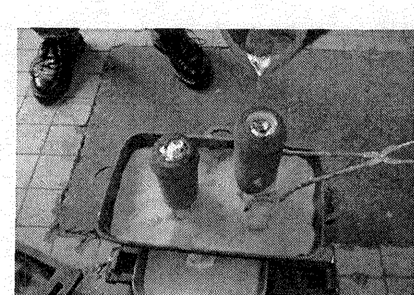

untreated

Figure 5 SLS of zirconium silicate – investment casting tests


preheating of the shells at 380° C


casting using an aluminum alloy at 750° C

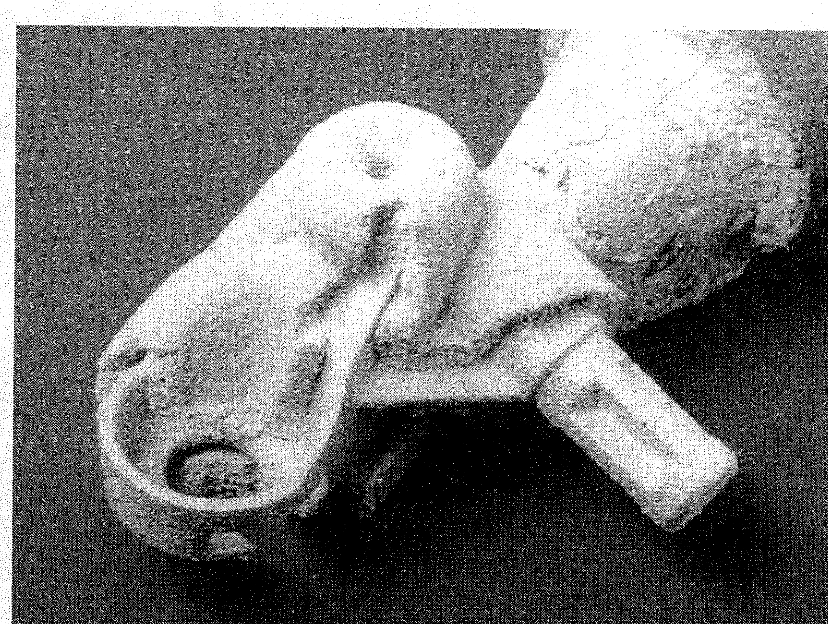

removal of the shell; cleaning; part is an articulated lever for the aerospace industry

Figure 6 Casting of the SLS shells

4 Summary and Outlook

Constructing the runner systems, combining the runner system with the parts, creating the shells and preparing the slice data for the SLS machine was done in two hours. Sintering the parts took about 20 h each, preparing the parts for investment casting (cleaning, preheating), casting the metal and removing the shells took additional 8 h. The overall lead time of two days compares favorably to a lead time of about two weeks required to produce the same parts using the conventional investment casting process. Thus, Selective Laser Sintering of zirconium silicate to ceramic shells for investment casting is an attractive alternative for rapidly manufacturing metal parts.

However, surface finish needs to be improved to satisfy industrial demands which poses a problem with the currently employed zirconium silicate; smaller grain sizes lead to agglomerates which hinder an even spread of new powder layers. Future work will thus include the finding of suitable ways to apply new layers of powder to the powder bed or the use of powders with more regular particle shape.

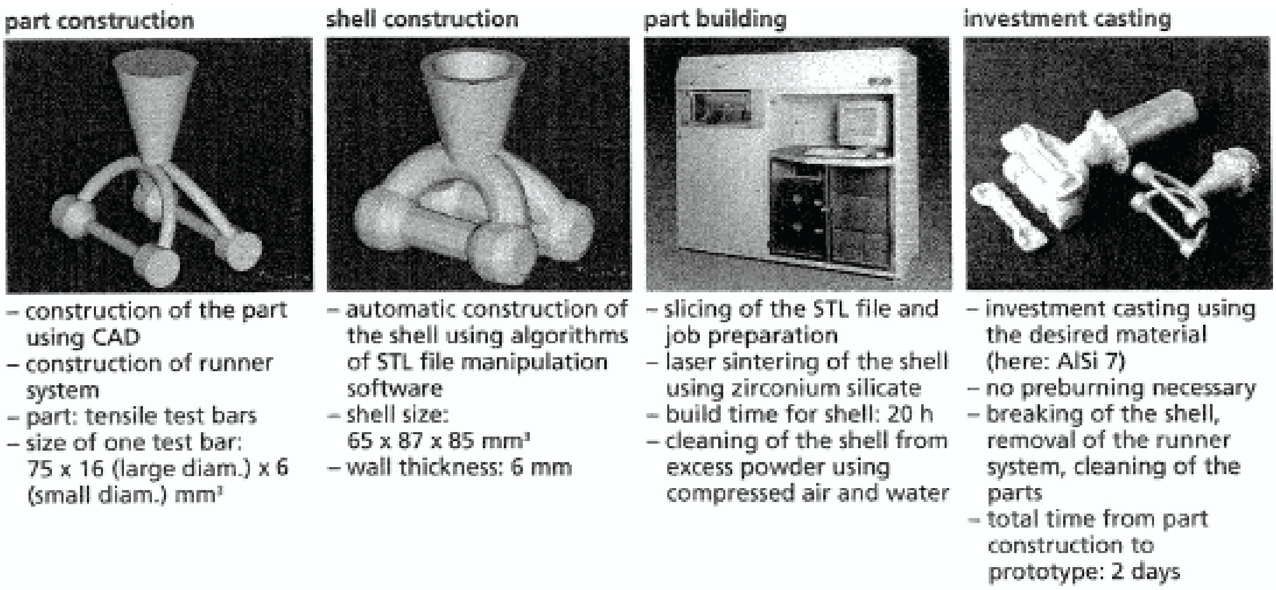


Figure 7 SLS of investment casting shells – rapidly to metal parts

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