Deliverable 6

Good Practice Guide

for

Detection and Prevention of Geometrical Deviations in Additively Manufactured Medical Implants

Project:

MetAMMI

Metrology for Additively Manufactured Medical Implants

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Content

1 Introduction

With the ability to produce patient specific medical devices on demand, the Additive Manufacturing (AM) technology is of growing interest for the medical sector. As the technology developed at a much faster pace than regulations or quality control, there is currently a lack of validated techniques to verify the finished parts. The intention of the MetAMMI project is to diminish these gaps and to increase confidence of the medical device industry in the AM technology.

In the frame of the MetAMMI project, various dense and lattice parts have been manufactured using different AM technologies and different materials. The parts have been characterized and typical geometrical deviations have been identified as well as their origin in the process chain. Based on this information, methods for the identification of typical deviations have been investigated.

The present Good Practice Guide summarizes the results of the investigations. It provides a short overview of the AM technologies and materials investigated. The different manufacturing processes and their advantages are described briefly, as more detailed information is available in literature.

In the subsequent chapter the test geometries printed by all the manufacturing partners involved in the project to be compared are presented and described briefly. Afterwards, deviations identified within the investigations of the project are listed and described in chapter 4, sorted by the manufacturing technology and material used for fabrication. Every deviation presented here is linked to possible detection methods and to a recommendation for its prevention.

Chapter 5 provides a quantification of defects detected in printed parts, based on investigations by X-ray Computed Tomography (XCT), while chapter 6 presents suitable measurement methods for the detection of defects. An analytical toolbox in section 6.1 allows to get a quick, rough overview on measurement methods and their general ability to detect certain types of typical defects. Parts with intended defects have been produced as described in section 6.2, to investigate several methods in detail. Three measurement methods and the related protocols for the detection of most typical defects are described in section 6.3.

Finally, chapter 7 describes recommendations for the prevention of deviations for the Binder Jetting process and for the Lithography-based Ceramic Manufacturing.

2 AM Technologies and Materials Investigated

Polymer, ceramic and metal parts have been manufactured by the AM processes subsequently described, starting from solid, powder or liquid feedstock.

2.1 Binder Jetting of Polymers & Ceramics

The Binder Jetting process is a powder based additive manufacturing technology, where a thin layer of flowable powder is applied on a build plate. The corresponding part information is transferred with an inkjet print head, which moves over the build plate and spits droplets of binding liquid onto the powder bed. When a layer is complete, the powder bed moves downwards, a new powder layer is applied and the whole process is repeated until the part is complete. Afterwards the part is in a green state and requires additional post processing, as removal of loose powder and support structures and additional temperature treatment. [1]

In general metal, polymer or ceramic based materials can be processed via Binder Jetting, but within the medical sector main focus is on the production of porous ceramic scaffolds for bone substitution applications. Typical materials used in this sector are Tricalcium phosphate or Hydroxyapatite. As the Binder Jetting process is relatively fast and cheap, it is also used to produce anatomic models made of polymer, e.g. for surgical planning.

This guide focuses on the for medical applications widely used materials TCP and PMMA parts.

Binder jetted parts typically feature rough surfaces, a porous bulk material and brittle behavior. Therefore, they are not well suited for load bearing applications, but offer advantages in the field of bioactivation and tissue ingrowth. [2] [3]

2.2 Laser-based Powder-bed Fusion of Metals

Powder material is applied with a recoating system layer-wise on a build plate and is melted locally in the area of the desired part cross section. Heat can be transferred to the powder bed either by a laser beam or by an electron beam. The laser beam is directed in an inert atmosphere using mirrors and generates heat in the powder bed by photons that are absorbed by the particles in the powder bed. Typically the part is connected directly to the build plate and need to be disconnected afterwards, e.g. by wire cutting as well as cleared from residual powder.[3, 4]

Besides metal also polymer and ceramic powder feedstocks can be processed with laser-based systems. However, focus in this project is on metal alloys typically used in the medical field, e.g. CoCr and Titanium alloys.

Parts produced with this technology show good densities but relatively rough surfaces and less accuracy compared to methods using liquid base materials, mainly due to the powdery feedstock and depending on build direction. In particular, the distribution of particle sizes has a significant influence on surface characteristics and achievable accuracy. Finer particles tend to lead to better accuracy and surface finish but are more difficult to process.

2.3 Material Extrusion of Polymers

Material extrusion (often referred to as Fused Deposition Modeling, FDM) of polymers is a widely used process. A solid starting material often in wire form is stored in a reservoir. It is forced through a nozzle and partially heated to liquify the material to be able to apply the resulting road on a build plate with a constant diameter and a constant velocity. The material becomes solid in the shape given by the moving nozzle and bonds to the previous road. To realize overhangs, support structures need to be build and removed again afterwards. [3]

Materials processed with this technology are typically thermoplastic filaments. Focus in the MetAMMI project was specifically on ABS. [3]

Parts produced by extrusion typically show round corners, no sharp edges and the single layers and roads are clearly visible on the whole part. [3]

2.4 Vat Photopolymerization of Polymers

For the fabrication of parts via vat photopolymerization or Stereolithography (SLA), a build platform is merged into a vat filled with liquid radiation-curable resins or photopolymers. The material is solidified locally and layer-wise by illumination of the desired part cross section with UV light, visible light or laser light thus starting the polymerization process. Light can be applied point wise via vector scan or via mask projection, which allows to illuminate and cure the whole part cross section in one layer at once due to the use of micro mirror arrays. The platform moves, a new layer of liquid polymers covers the former solidified one and the process is repeated until the part is finished. Subsequent post processing occurs by removing residual liquid polymer on the part and by post-curing them under UV light. [3]

The application of this process is limited to photopolymers and composite materials, which can form for example metal or ceramic parts after sintering.

Vat photopolymerization typically shows highest part accuracy and surface finish among the AM technologies. Of disadvantage is the aging of the material, resulting in degraded mechanical properties over time. [3] One of the biggest challenges is that the overhanging parts need support structures and that the part need to attach to the build platform with the support. Since the support structure is made of the same material as the actual parts, those need to be removed mechanically.

2.5 Lithography-based Ceramic Manufacturing of Ceramics

Lithography-based Ceramic Manufacturing (LCM) technology is based on the vat photopolymerization principle and enables the layer-wise manufacturing of ceramic parts featuring material properties comparable to conventionally manufactured high performance ceramic parts.
Solid content suspension of ceramic particles in a liquid photopolymerizable resin is selectively exposed to ultraviolet (UV), visible or infrared light. Individual mirrors of a digital mirror device can be selectively turned on and off illuminating/exposing the entire contour of each cross section of the 3D object at once and thus curing the resin in defined areas by photopolymerization. Each layer of the ceramic suspension is applied via a blade on a rotating material vat. The build process occurs bottom-up, when the build plate is moved to the vat leaving a gap of one layer. After curing, the solidified layer which is attached to the build plate is separated from the material vat by tilting it away from the build plate. The layer is finished and the sequence repeats, to finally produce the so-called green part, by a composite of ceramic particles in a polymer matrix. To obtain dense ceramic parts, the polymer is removed from the green part by thermal decomposition. Subsequent high temperature treatment leads to densification of the 3D object by sintering of the ceramic particles.[5] [6]

Generally, all sinterable materials in powder form can be processed using LCM technology. Focus within MetAMMI project is on ceramic biomaterials, especially Alumina, β-Tricalcium phosphate (β-TCP) and Alumina Toughened Zirconia (ATZ).

The resulting ceramic parts typically show high densities, a smooth surface and homogeneous material distribution. Due to the sinter process, shrinkage of the part occurs, which needs to be considered during design.

3 Part Geometries Manufactured

The following part geometries have been manufactured within the MetAMMI project. The images show the nominal condition defined by the STL files used for manufacturing and can be compared to the defective parts presented in chapter 4. Dense and lattice parts have been chosen for manufacturing, including circular features, holes or fine features.

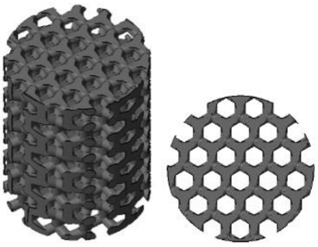


Figure 1: Cylindrical Scaffold (top view on the right)

A cylindrical scaffold based on a Wurtzit structure was manufactured to analyze, which type of defects typically emerge in fabricated lattice parts. In the original file the scaffold has a nominal diameter of 6.8 mm, a height of 8.5 mm and a strut thickness of 0.5 mm in diameter. This filigree structure could not be realized with all technologies. If necessary, it was scaled up. The geometry was also used to introduce intended typical defects to study the suitability of different measurement methods.

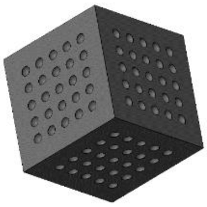


Figure 2: Calotte Cube

A calotte cube was fabricated by all partners in a size of 10 x 10 x 10 mm and calottes (i.e. inner half spherical shape) with a diameter of 0.8 mm. This part is also commercially available and used for testing of µCT, µ-optical and µ-tactile measuring systems by measuring center to center distances. [7]

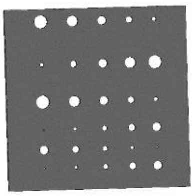


Figure 3: Cube with designed Defects

A square part with a side length of 20 mm and a thickness of 4 mm was fabricated with cylindrical defects included in terms of channels and calottes with diameters in a range of 0.2 – 2 mm. Flatness, straightness and right angles can be determined as well as concentricity of cylindrical defects.

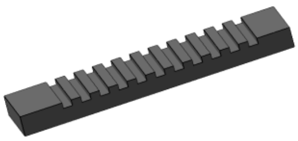


Figure 4: Step Gauge

A step gauge which is prone to warpage was manufactured with a length of 55 mm. Measuring of flank-to-flank distances allows to draw conclusion on the part deformation.

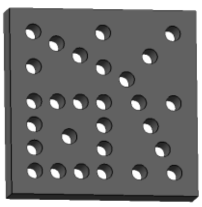


Figure 5: Hole Plate

Another standard object is a square hole plate with a side length of 48 mm, a thickness of 8 mm and hole diameters of 4 mm. Form and center-to-center distances of the holes can be measured.

4 Typical Deviations in Manufactured Parts

Parts printed within the project have been investigated by XCT. Defects identified are presented in this chapter, as well as possible measures for detection and prevention.

4.1 Deviations in Parts made by Binder Jetting

Parts resulting from this process are typically very porous and depending on the application, post processing like infiltration of polymer parts or sintering of ceramic parts is often required to improve mechanical properties.

Shrinkage

Shrinkage of parts is a common phenomenon in processing of ceramic materials. It results from heat treatment of the part after printing, which is required to achieve sintering (compaction and diffusion) of the particles and sufficient part stability. The shrinkage is typically not homogeneous and varies in x-y (plane of the build plate) and z direction (height of the part).

Shrinkage was observed up to 20 % in the printed ceramic scaffolds and the shrinkage in z direction was typically higher than in x-y direction, which can be related to the unisotropic properties of the green part. For the polymer parts, shrinkage was around 4 % in x-y direction and smaller in z direction (≤ 2%).

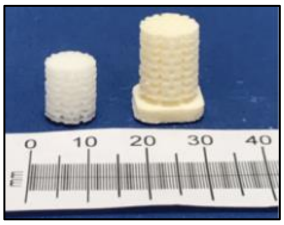


Figure 6: Ceramic part sintered (left) & as printed with support (right)

Measures for Detection

Shrinkage is detectable by nominal actual comparison via XCT or by simple measurement of the outer contour with a caliper.

Measures for Prevention

The degree of shrinkage depends on the properties of the powder used for printing, the specific printed geometry as well as temperature and time for debinding and sintering. The correlations are described more detailed in section 7.1. The shrinkage factor needs to be known, e.g. from test prints and considered already during design by upscaling the STL file.

Warping

From Figure 7, warpage in dense ceramic cubes is visible, introduced by the temperature treatment of the parts. The planes of the cube are curved to the center of the part. The XCT image in Figure 8 shows in addition more densification of the cubes at the edges than in the center, as the edges reaches higher temperatures earlier and show an increased sinter activity. This avoids the release of gases from the center of the part and lead to unisotropic shrinkage and deformations.

On polymer parts, warpage was not observed on dense massive parts, but on thin bars during drying process.

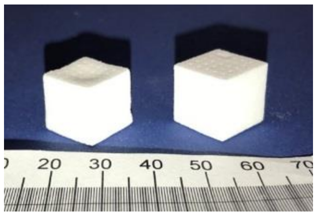


Figure 7: Warping on ceramic calottes

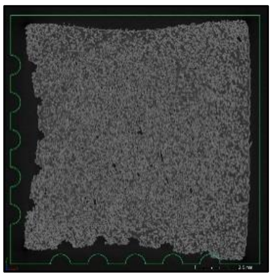


Figure 8: XCT image of ceramic calotte

Measures for Detection

Warpage of the parts is often visible by eye.

Measures for Prevention

Warpage of parts can usually be avoided by an adjusted temperature treatment, especially by a longer debinding time and a slower heating and cooling rate (see also section 7.1).

Porosity

A high amount of residual porosity is typical for the Binder Jetting process and desired for instance for bone substitution parts. Porosity in printed parts has been observed around 34 % in sintered ceramic parts and around 46 % in printed polymer parts.

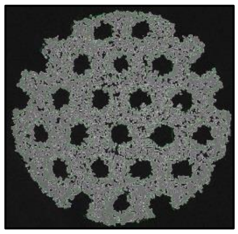


Figure 10: Porosity of ceramic part

Measures for Detection

While porosity analysis by XCT was possible to be performed on parts printed by other AM technologies, it was not possible for parts produced by Binder Jetting. For the determination of the amount of porosity in the solid both Archimedes and measurement with gas pycnometer are suitable measurement methods. For lattice geometries on the other hand, the gas pycnometer is more reliable as in case of Archimedes method, remaining liquid in the designed channels can influence the measurement.

Measures for Prevention

Residual porosity is typical for this process. The amount of residual porosity depends on the powder properties of the feedstock (e.g. particle size, packing rate, flowability) and the heat treatment (see also section 7.1).

Residual Powder

Residual powder in the part is a typical phenomenon in powder-based processes. Within the frame of the project, it has been observed in lattice polymer parts printed by Binder Jetting. It has not been observed on the ceramic lattices printed by Binder Jetting. For polymer parts, a hexanol-based solvent has been used as binding liquid, which leads to a connection of the particles due to a chemical reaction and evaporates afterwards. The vapor seems to affect also surrounding powder and impedes the subsequent removal of powder in cells of lattice geometries as visible in Figure 11 and Figure 12. Extensive cleaning with compressed air and thin tools allows to remove the powder off the outer regions of the geometry, but not in the center.

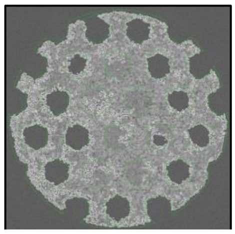


Figure 11: Residual powder in polymer part (top view)

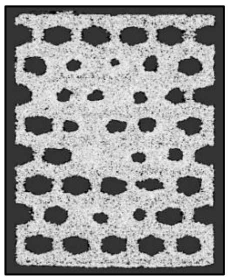


Figure 12: Residual powder in polymer part (side view)

Measures for Detection

Residual powder in lattices as presented are visible by eye or optical microscope.

Measures for Prevention

The interaction of material and binding liquid needs to be considered during design of the part as well as accessibility for cleaning tools.

Missing Features

Both ceramic and polymer parts printed by Binder Jetting showed missing areas in the outer region of the lattice geometry. Especially the ceramic parts can easily be damaged during handling, as they are very fragile before final heat treatment.

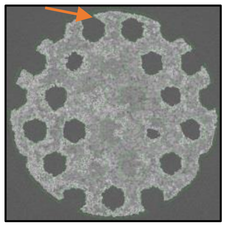


Figure 13: Missing fine strut in polymer part

Measures for Detection

Missing areas in the outer regions are visible by eye or under optical microscope. For the detection of broken or missing struts in the center of the part, methods for the investigation of the total volume as XCT becomes necessary.

Figure 14: Scaffold with support structure

Measures for Prevention

The minimum possible feature size of the system used for printing has to be considered during design. In addition, careful handling during cleaning of fragile parts is necessary, especially for printed ceramic parts, which have a low mechanical stability prior to the sinter step. The design of a support structure as visible in Figure 14 was required to allow handling of the lattices. It was removed subsequent to the sinter step.

4.2 Deviations in Parts made by Laser-based Powder-bed Fusion

For laser-based Powder-bed Fusion the quality of the final parts is mainly influenced by the properties of the powder, powder bed temperature, laser energy input as well as the scan strategy. The combined effect of the major process parameters on defect formation can be expressed by the energy density E [J/mm³], which is defined as follows according to Zhang et al. [8]:

$$E = \frac{P}{v * h * t}$$

The relation between laser power introduced (*P*), scan speed (*v*), hatch spacing (h) and layer thickness (*t*) supports the selection of appropriate build parameter to reduce defects in the printed parts. Defects and deviations which typically occur in parts produced by LB-PBF are presented in the following.

Pores and Lack of Fusion Defects

Typical defects for parts produced by laser-based Powder-bed Fusion are spherical pores and lack of fusion defects.

Randomly distributed spherical pores (see Figure 15) can result from gases entrapped in the melt pool due to a fast solidification rate. They are difficult to avoid and can increase with a lower packing density of the feedstock. [8]

Lack of fusion defects on the other hand are characterized by an elongated shape and represent poor bonding defects with unmelted metal powder as a result of insufficient molten material during the solidification process. This can occur for instance, when the laser energy is too low or the width of the melt pool is too small leading to insufficient overlaps between scan tracks. A low laser energy can also lead to insufficient penetration depth which leads to poor interlayer bonding. These defects typically occur between laser tracks or between layers. [8]

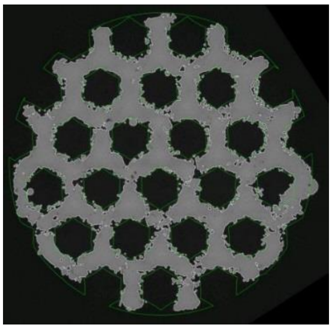


Figure 15: Scaffold, Titanium alloy

Measures for Detection

Pores can be detected by means of metallographic investigations during parameter studies. For non-destructive testing, volumetric methods as XCT are suitable or also acoustic methods as presented in chapter 6.3.3.

Measures for Prevention

The development of pores can be attributed to scan parameters but also related to the quality of the feedstock, i.e. internal voids in the powder raw material can cause defects. Powder can be produced using different methods as for instance gas atomization (GA) or Plasma Rotating Electrode process (PREP). According to Taheri et al. [9], less interlayer porosity occurs with PREP powder. Also, packing density of the powder has an effect on porosities in the final part as well as any impurities.

The scan strategy can influence the formation of defects as well. While unidirectional input of laser energy more frequently generates incomplete fusion defects, cross hatching on the contrary leads to a more balanced energy input, preventing accumulation of defects. [8]

Also preheating of the powder can reduce porosities in the final part as presented in Figure 16 and Figure 17. Preheating was carried out at 360°C. [10]

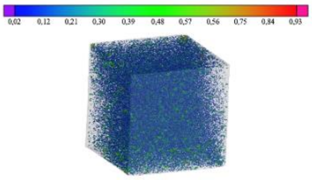


Figure 16: XCT porosity analysis on cube printed without preheating

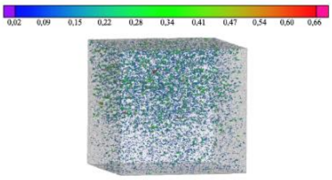


Figure 17: XCT porosity analysis on cube printed with preheating at 360°C

Cracks/ Distortion

During the manufacturing process, metal powders experience rapid melting and rapid solidification with cooling rates up to 10°8 K/s. This results in a great temperature gradient and large residual stresses in fabricated parts. High temperatures and residual stress causes cracks and crack propagation in final parts as well as distortions. [8]
Cracks have been observed in a calotte cube as presented in Figure 18 and distortions for example in flat geometries as in the hole plate presented in Figure 19. Deviation from the nominal part are indicated in red and blue.

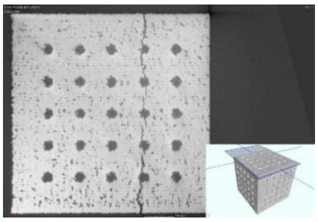


Figure 18: Calotte Cube, Titanium alloy

Measures for Detection

Surface cracks are detectable by eye or by optical microscope. For internal defects acoustic methods can be taken into account. In addition, nominal/actual investigation by XCT is suitable to visualize distortions.

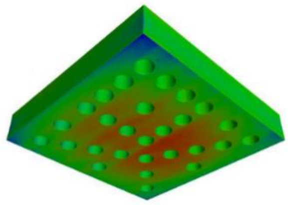


Figure 19: Hole plate, Titanium alloy

Measures for Prevention

Preheating of the powder bed helps to reduce thermal stresses in the parts, while additional support structures can reduce bending.
The effect of preheating the powder has been investigated and is visualized in Figure 20. The twin cantilever was made of Titanium alloy (TiAl6V4) with no preheating (a), preheating up to 230°C (b) and 360°C (c) which leads to significantly less deformation. [10]

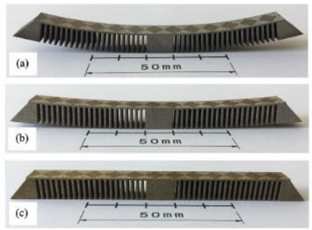


Figure 20: Effect of preheating the powder bed

Roughness

Without further post processing, the surface of parts produced by LB-PBF appears rough as visible in Figure 21. As upward facing features on the build plate solidify against air, they are likely to show better surface properties as down or sideward facing features, which solidify against powder. [3]

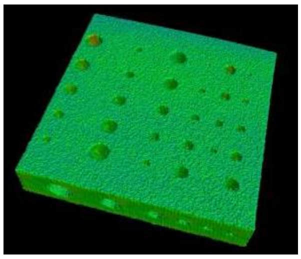


Figure 21: Cube with defects, Titanium alloy

Measures for Detection

For the determination of surface roughness, several measurement methods as listed in chapter 6.1 are available.

Measures for Prevention

Roughness can be reduced especially with postprocessing methods as sand blasting.

Shrinkage line

When structures of the parts are merging after building separately in the powder layers, a line may appear on the part, corresponding to the layer(s) in which the coalescence has happened, hereby creating surface tension in the melt pool and a deformation visible on the solidified part.
As visible in Figure 22, the shrinkage line (circled in blue) corresponds to the merging of the lateral arch pointed in blue.

Figure 22: Shrinkage on a spinal implant built in the direction $\vec{Z}$

Measures for Detection

The detection is observable after production. Detection may thus happen during early-stage process qualification for a given product.

Measures for Prevention

The visibility of such shrinkage lines can be reduced with adequate postprocessing methods such as sand blasting or chemical etching, this is illustrated in Figure 23.
Furthermore, this type of defect can also be addressed by changing the part orientation. With recent progress on process simulation, the detection and correction of these defects will likely be possible during the pre-processing steps.

Figure 23: Spinal implant after post processing

4.3 Deviations in Parts made by Material Extrusion

Critical to the quality of the parts printed by Material Extrusion is the composition and homogeneity of the filament used to create the single layers as well as the deposition procedure. Influencing parameters are the layer thickness related to the nozzle size and extrusion temperature as well as the velocity during deposition and the printing pattern with raster angle and width. Typical defects observed on the part printed within the frame of the project are summarized below.

<u>Under Extrusion and Voids</u>

Unintended gaps can occur between the contour and infill lines, if not enough polymer is extruded during filling of parts due to the parameter combination chosen for printing. As a result, under extrusion occurs as visible in Figure 24. [11]
Incomplete filling can also lead to voids as presented in Figure 25, resulting from turn points in a raster pattern. The size of voids thereby depends on the width of the roads, the thickness as well as the angle between raster and perimeter curve. Kim et al. [12] reported by trend more voids in the upper region of a part, as the lower part of a printed part is more compacted by heat and pressure of the material in the following layers which leads to larger neck growth between roads and reduced/ smaller voids.[12]
Excessive application of extruded material on the other hand can lead to defects described as over-extrusion which leads to a loss of geometry. This has not been observed on the parts printed within the frame of the project.

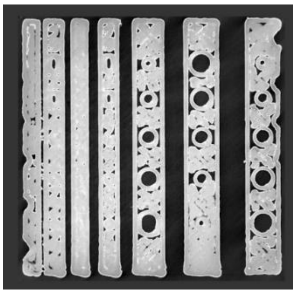


Figure 24: Cube with defects showing under-extrusion

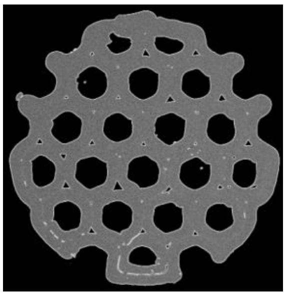


Figure 25: Scaffold, ABS with voids

<u>Measures for Detection</u>

Under extrusion and voids are often visible by eye and optical microscope.

<u>Measures for Prevention</u>

Voids can be reduced with an adjustment of the deposition pattern to the part geometry. Deposition methods, in which the roads follow the contour of the part for instance increase the build time but lead to less voids. Raster deposition in zig zag pattern on the contrary decreases the build time while leading to more voids due to the high amount of turning points. On overlap between roads can reduce voids at turning points, while too much overlap or a too high negative gap respectively leads to flooding of materials and over extrusion. A good quality of the filament in terms of uniform diameter is as well crucial for the development of voids. [12] [13]

<u>Shrinkage/ Distortion</u>

No significant shrinkage of the outer geometry has been observed but struts thinner than designed in printed scaffolds as presented in chapter 5. According to Turner et al. [14] the error in accuracy in final printed parts by Material Extrusion arises from shrinkage during cooling and solidification or warping as uneven heat distribution creates internal stresses. Stresses produced by thermal gradients can also cause delamination of roads or cracks. Thermal gradients can result for instance from the deposition of the hot melt on a previously deposited partially printed cool part.

<u>Measures for Detection</u>

Caliper, XCT

<u>Measures for Prevention</u>

To reduce thermal stresses in the parts and assure proper bonding between roads, a controlled environment temperature is recommended.

Depending on the geometry, support structures can prevent distortions or warping. The degree of shrinkage of the material due to solidification and cooling can be investigated and compensated in the part design.

Roughness

Parts produced by Material Extrusion typically show rough surfaces due to the deposition of single roads. The single layers are clearly visible in the final part as demonstrated in the XCT image in Figure 26. Filament width as well as the orientation of the part during printing affect the final surface quality.
In addition, the first layer can adapt the roughness of the build plate, while the top layer is ridged due to the single roads. The removal of support structures can lead to fine holes on the surface. [13]

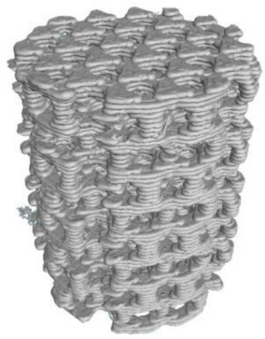


Figure 26: Scaffold, ABS

Measures for Detection

For the determination of surface roughness, several measurement methods as listed in chapter 6.1 are available.

Measures for Prevention

Surface properties can be improved by smaller filament diameters or post processing methods using chemicals as acetone with water to seal the surfaces and make them impervious to water.

Stringing/ Oozing

Another defect typically reported for filament based parts is stringing or oozing, which occurs when traces of small strings of polymer are left behind the nozzle while it is not extruding material and moving to a new starting point. [11]

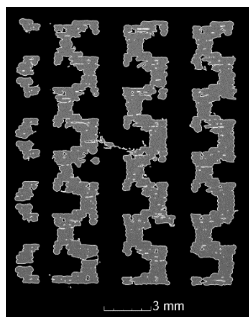


Figure 27: String in scaffold, ABS

Measures for Detection

Visual inspection supported by optical microscope.

Measures for Prevention

An adjusted nozzle temperature and increased retraction can generally avoid stringing, which occurs when the temperature is too high or retraction (pull back of filament) not strong enough. In addition, the probability of occurring is higher in designs with small gaps.

4.4 Deviations in Parts made by Vat Photopolymerization

Vat photopolymerization in terms of Stereolithography represents the earliest AM technology, which has been widely investigated. Main advantages of this technology are the achievable accuracy and surface finish. Quality influencing parameter are for instance scanning speed and laser spot diameter, which have an influence on line width and cure depth and therefore affect the vertical and lateral resolution.[15] Typical deviations reported in literature are related to shrinkage, which occurs as the volume occupied by the monomer molecule is larger than the volume of the reacted polymer and also to distortion resulting from thermal stresses as a result of heat release due to chemical reactions. Low scan speeds can increase these deviations as they lead to higher temperatures and reaction rates.[3] [12]

Shrinkage and distortion have not been observed to a significant extend within the project. Deviations observed were broken areas in very filigree structures as well as an uncured area in one part.

<u>Missing Spot & Broken Regions</u>

In one of the scaffold geometries printed a missing spot was observed which propagates from the top to the middle of the geometry as visible in the top view in Figure 28 and the side view in Figure 29. This is probably caused by contaminations in the vat like old resin.
In addition, broken features were observed in the very filigree parts as a result from detaching them off the build plate. This can be avoided by using support structures, while the removal of support structures from very fragile parts is challenging as well.

Figure 28: Scaffold, Dental resin (top view)

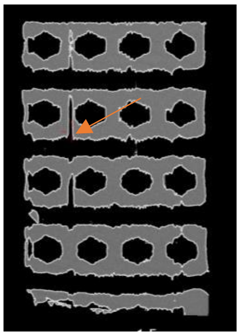


Figure 29: Scaffold, Dental resin (side view)

<u>Measures for Detection</u>

Broken areas can be detected by visual control and missing spots/cracks can be detected in the transparent material using optical microscopy.

<u>Measures for Prevention</u>

To avoid uncured areas the vat has to be free of contaminations. Broken regions can be avoided by adjusted support structures or an upscaling of the design.

<u>Residual Fibers</u>

Within the cells of the printed scaffolds, residual fibers have been observed as visible in Figure 30, which can be a result of residuals of the resin in the cells. During cleaning of the parts, it might be that those do not solve to the alcohol or solve partly. When the parts are removed from the alcohol and existing alcohol has vaporized these residuals might start flow and form extra fiber looking structures. Post-curing process then cures these residuals together with the part, forming deviations. In addition, laser scattering in the photopolymer or fiber types of extra particles in the resin or residuals for example from cloth used to dry the parts might cause similar fiber type deviations.

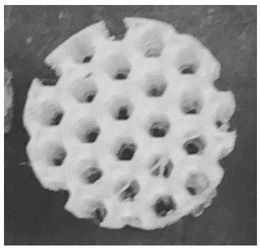


Figure 30: Scaffold, Dental resin with fibers in cells

<u>Measures for Detection</u>

Visual inspection supported by microscopy.

<u>Measures for Prevention</u>

Moving the parts or the liquid in cleaning phase. Avoid small holes etc. in the design phase.

4.5 Deviations in Parts made by Lithography-based Ceramic Manufacturing

Typical defects observed in ceramic parts produced by LCM were predominantly delaminations and overall thicker struts in case of lattice geometries, which are described in the following.

Cracks and Delaminations

Both lattice and dense parts printed within MetAMMI showed delaminations and cracks caused by delaminations as visible in the Alumina plate in Figure 31 as well as in the scaffold presented in Figure 32.

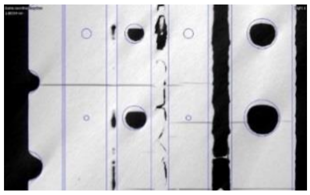


Figure 31: Plate with defects, Al_2O_3

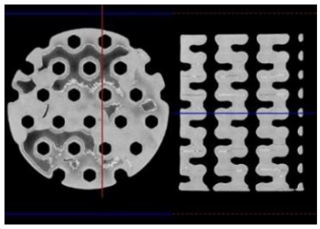


Figure 32: Scaffold with delaminations, TCP

Measures for Detection

Translucent parts can be inspected for cracks by checking the light transmission. Cracks appear as dark lines and can be detected quickly and easily. In addition, an infiltration test with a fluorescent dye can be used to detect cracks. The latter technique is particularly advantageous for highly opaque materials with low light transmission and scaffold constructions. XCT is another possible technique.

Measures for Prevention

The cracks indicate delaminations. Several measures can be taken to prevent delaminations in the component. An adjustment of the printer parameter settings (I), an adjusted cleaning (II) and thermal post-processing (III) or design adjustments (IV).
Ad (I): Higher exposure energy, lower layer thickness, slower tilting and longer waiting times are main parameters that can reduce the risk of delaminations.
Ad (II): A sufficiently small amount of solvent should be used for cleaning, followed by complete drying of the components with compressed air.
Ad (III): Temperature programs can be adapted by optimizing or extending heating rates or holding times.
Ad (IV): Rounding or chamfering on sharp edges and corners reduces the risk of delaminations. Sharp edges and corners induce local stress peaks and thus promote crack formation and propagation.

Detachment Defect

The defect presented in Figure 33 was observed at the bottom of the part, were it was attached to the build plate during printing. The detachment of the part from the building platform or adhesive layer leads to surface cracks.

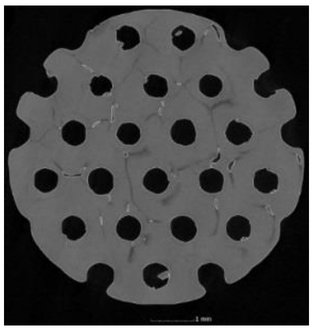


Figure 33: Scaffold, TCP with detachment defect

Measures for Detection

Defects on the bottom of the component can be observed optically using a light microscope. Also, more advanced techniques such as XCT, or laser scanning can be used.

Measures for Prevention

The component can be carefully separated from the building platform by using a sharp blade. The adhesive layer of the component can then be carefully removed with the blade too. Correct adjustment of the adhesive layer simplifies the separation of the components. The thickness of the adhesive layer plays an important role, which can vary depending on the design of the component.

Also, the use of support structures as well as their design can have an effect on the surface quality and the presence of defects at the sides facing the building platform.

Dimensional Variation

Cells were observed to be smaller than designed due to overall thicker struts, when comparing XCT data to the STL file (green line in Figure 34). Due to light scattering, over-polymerization occurs, which depends on the material and the exposure energy. In addition, uncured slurry adheres to the surface of the printed green body. Especially in fine channels, slurry can accumulate due to capillary forces, which can lead to increased lateral over-polymerization and partial clogging of the channels.
During cleaning, the excess uncured slurry is removed from the surface. However, fine channels with high aspect ratios are more difficult to clean; also the cleaning status cannot be monitored for the inner surfaces.
For very fine elements deviations can also arise from the pixel-based exposure as the free-form area from the underlying STL data can only be approximated using square elements of a given pixel size.

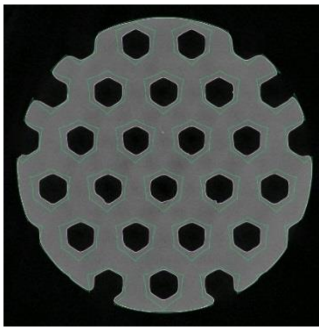


Figure 34: Scaffold, TCP showing dimensional variation – CAD (green line) compared with sintered part

<u>Measures for Detection</u>

Dimensional variation can be observed optically using a caliper and/or light microscope for the elements at the outside. For the overall structure XCT can be used. Weighing the samples is a possible approach, however using this technique only larger deviations can be detected.

<u>Measures for Prevention</u>

Each material is characterized in terms of curing depth and over-polymerization. Based on these data, the appropriate exposure energy is selected to ensure sufficient curing depth and minimal over-polymerization. In addition, the parameter setup can be adjusted by setting a contour offset to compensate lateral over-polymerization. In order to compensate over-polymerization in z-direction, another parameter, the “curing depth compensation”, can be adjusted.
By increasing the temperature of the slurry during printing, the viscosity can be lowered, reducing channel clogging and thus over-polymerisation.
For very fine structures, the dimensions must be matched to the pixel size of the machines. For optimum resolution, channels and struts should be a multiple of the pixel size. If this is not the case, irregular pixel sequences can occur, leading to dimensional variations.
In particular, this applies to very fine, high-resolution structures with regular strut and channel sizes.

Shrinkage

Shrinkage of the parts is an inevitable deviation occurring due to sintering of ceramic particles. Depending on the solids loading of the suspension, the parts shrink by up to 29 % during sintering. For this reason, the design of the components is scaled up by a defined shrinkage compensation factor. If this factor is not or only inaccurately defined, dimensional deviations occur. Regarding large components, slight inaccuracies in the shrinkage compensation factor can lead to larger dimensional deviations compared to small components.

Measures for Detection

Dimensional accuracy and hence shrinkage can be determined with a caliper and/or light microscope by measuring the green and sintered state of the component in X-, Y- and Z-direction. For more complex components, where certain structures are not visible for a standard measurement, CT or a laser scanner can be used. The compensation factor is calculated from the determined shrinkage.

Measures for Prevention

The shrinkage can be compensated by scaling the design with a shrinkage compensation factor directly in the software of the printer.

5 Quantification of Geometrical Deviations in AM Technologies

The cylindrical scaffold introduced in chapter 3 was manufactured with every AM technology involved in the project and allows to compare and quantify typical deviations in additively manufactured parts. Every manufacturer chose suitable build parameter for this type of geometry, which required an upscaling of the original STL file in some cases as visible from Figure 35.

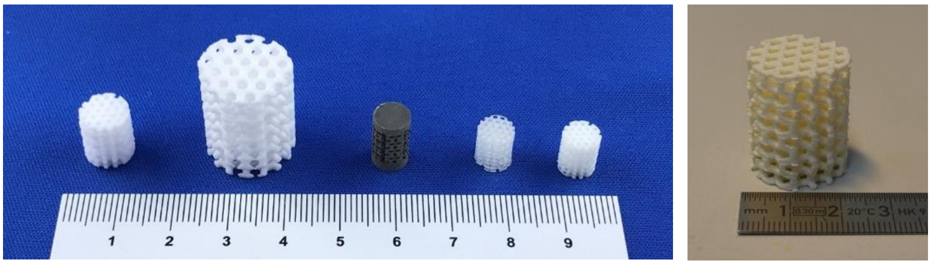


Figure 35: Scaffold produced by BJ (TCP/ PMMA), LBPBF (CoCr), SLA (Resin), LCM (TCP), FDM (ABS)

Parts produced by Binder Jetting had to be scaled up by the factor of 1.5 for TCP and 2.5 for PMMA and the part produced by Material Extrusion by a factor of 3 to realize the filigree structure. Afterwards, the parts were investigated by XCT measurement (Figure 36) and analyzed regarding deviations in the outer geometry, deviation in overall geometry and deviation in strut thicknesses.

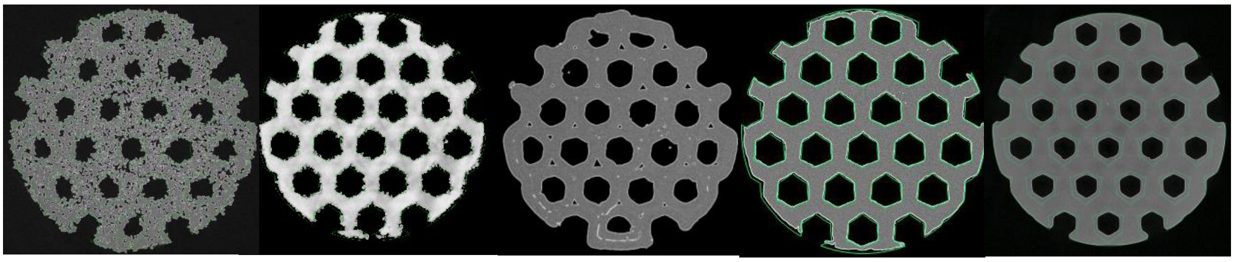


Figure 36: XCT images of Scaffolds printed by BJ (TCP), LBPBF (CoCr), FDM (ABS), SLA (Resin), LCM (TCP)

The outer geometry was determined by a scaling factor based on the outer contour of the scaffolds, i.e. measurement of the outer diameter and the height, and comparison with the nominal model (i.e. STL model). The overall geometry (inner and outer) was evaluated by an actual/nominal comparison of the measured/produced part with the nominal STL model of all parts. The deviation distribution of each measured part was determined. Absolute deviations in µm at 50 % and 90 % of the total deviation are used as quantities to characterise the AM parts.
According to the results shown in Figure 37, no significant overall shrinkage has been observed in parts produced by FDM and SLA, while slight shrinkage has been observed in polymer parts produced by Binder Jetting and metal parts produced by PBF-LB. Due to required sintering, most shrinkage occurs in parts made of ceramic. For parts produced by LCM, shrinkage was compensated in design finally resulting in slightly greater dimensions.

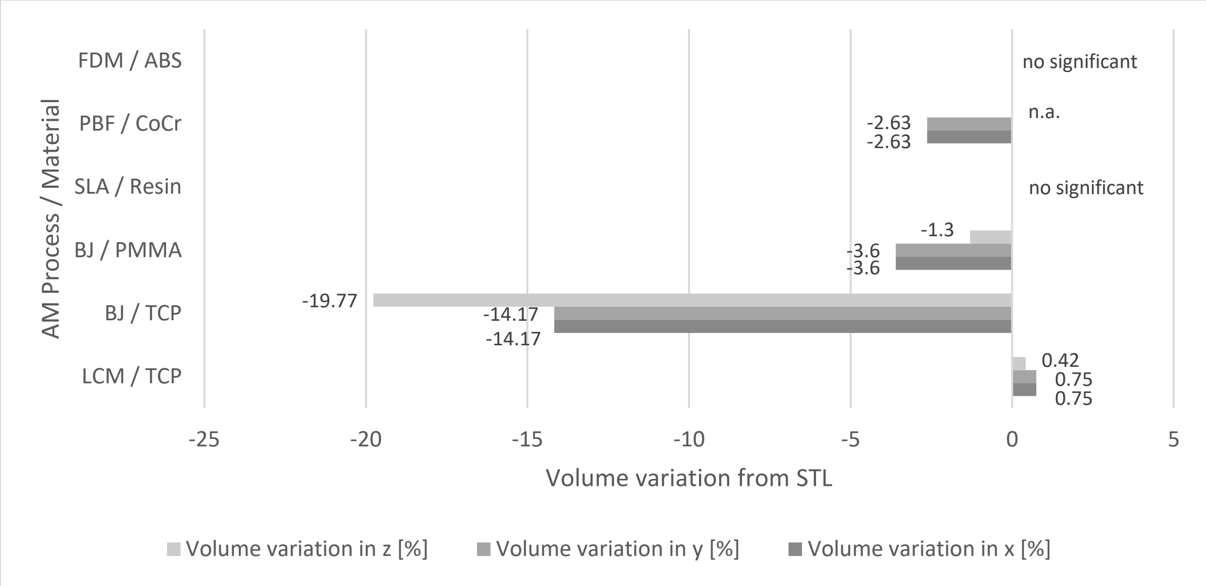


Figure 37: Deviation in outer geometry

Comparing the overall geometrical deviation (see Figure 38), the highest accuracies have been achieved with SLA process, followed by PBF- LB and LCM. Parts produced by FDM and BJ showed most internal defects, irregular contours as well as remaining powder leading to overall greater dimensional deviations.

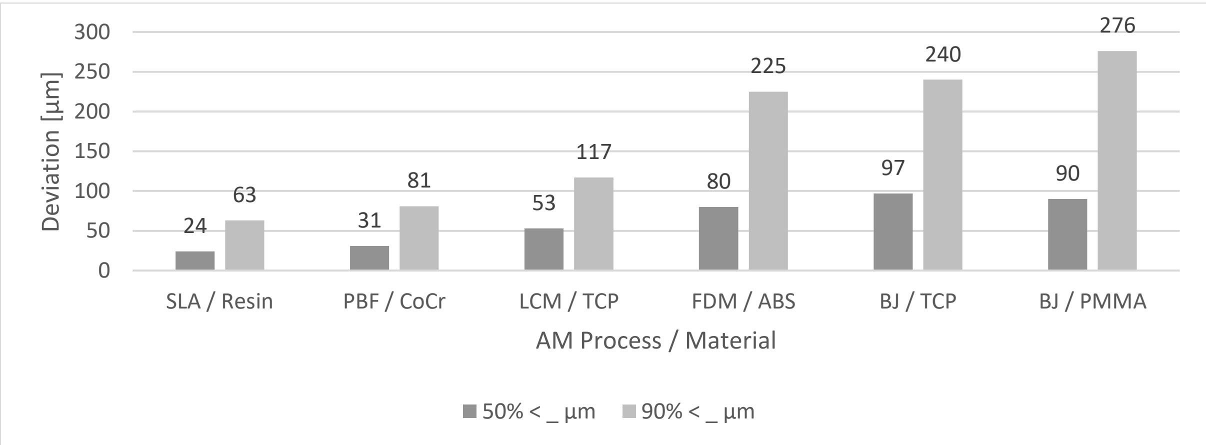


Figure 38: Overall geometrical deviation

For the porosity measurements, an algorithm implemented in the commercial software for XCT visualisation and measurement VG Studio Max 3.0 was used. The algorithm searches for pores within a determined - by the user – range of pore diameters and provides the percentage of the pores relative to the total volume. No significant porosity has been observed in the part produced by SLA and low porosities in parts produced by PBF-LB and LCM (probably due to delamination defects). The high residual porosities for parts produced by Binder Jetting have been investigated by Archimedes method, showing approx. 46 % porosity for the polymer parts and 34 % for the printed and sintered ceramic parts (see Figure 39).

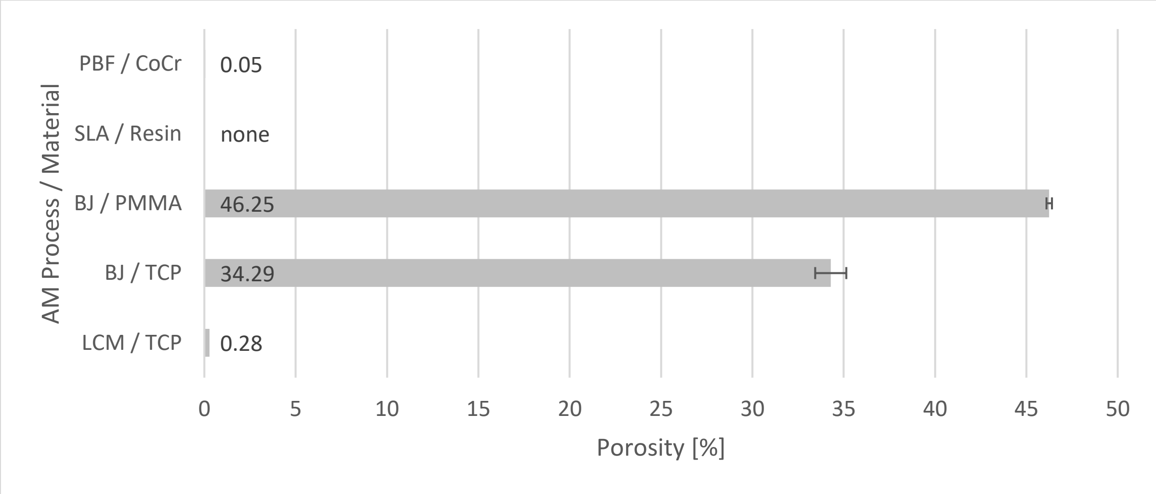


Figure 39: Porosity

The strut thickness was calculated based on geometrical measurements (of cylinders) in the struts. Diameter of the strut cylinder was measured in different regions of the cylindrical scaffold and the averaged diameter is presented in Figure 40 in comparison to the nominal diameter of the STL file.

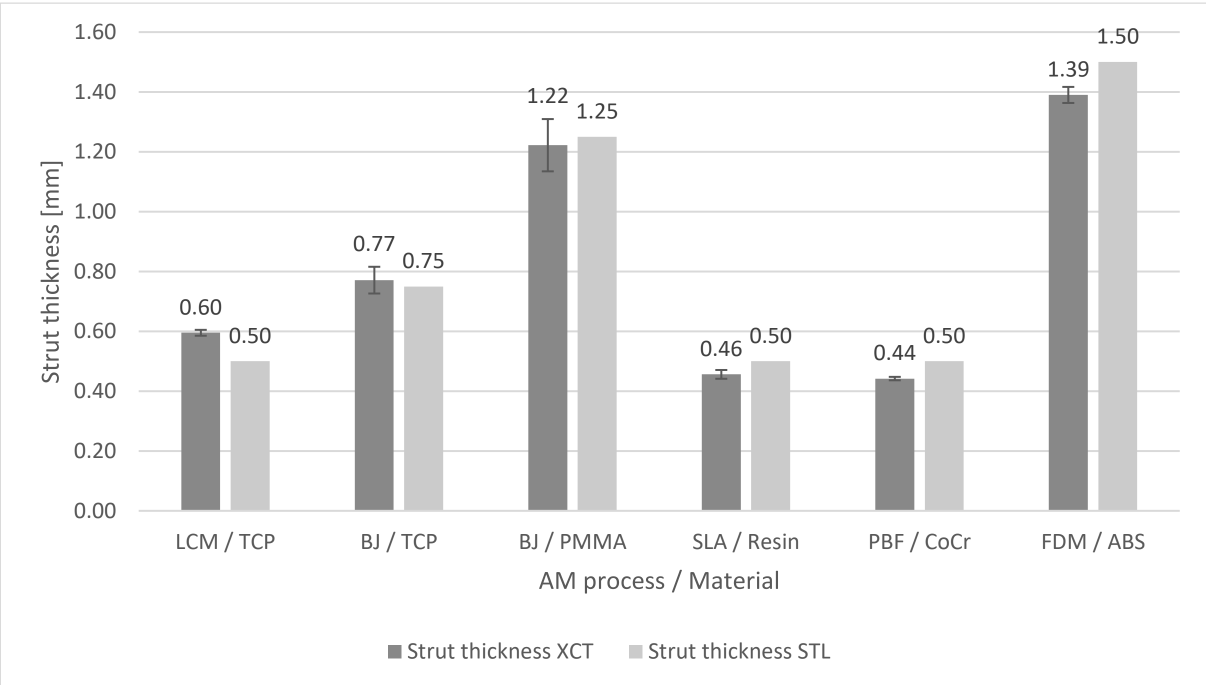


Figure 40: Strut thicknesses

Figure 40 shows overall thicker struts for parts produced by LCM, no significant variations in strut thicknesses for parts produced by Binder Jetting and overall thinner struts for parts produced by Stereolithography, PBF-LB and FDM.

6 Recommendations for Identification of Deviations

This chapter presents possible non-destructive measurement methods and their suitability to detect certain types of deviations in additively manufactured parts described before in chapter 4.
An analytical toolbox in section 6.1 provides a quick overview on the different measurement methods investigated in the project and for the detection of which defect category they are basically suitable. In addition, the toolbox allows to roughly compare possible resolution, sample requirements and efficiency of the different measurement methods.
Section 6.2 shows the design of parts with known defects, which have been manufactured for the development of measurement protocols as well as the defect verification by XCT investigation.
Defect detection by weighing, by Archimedes methods and by resonant acoustic testing are presented in detail in the following section 6.3, giving three measurement protocols.

6.1 Analytical Toolbox

Table 1: Analytical Toolbox

		Volumetric Methods					Surface Methods						Density & Porosity		Others	
		XCT	THz-CT	Thermography	Ultrasonic Testing	Resonant Acoustic Testing	CMM	3D Laser Scan	Fringe Projection	Focus Variation	Confocal Microscopy	Stylus Instrument (tactile)	Archimedes	Pycnometer	Permeability	Weight Measurement
Detectable Defects																
Defect Categories	**Example**															
Dimension Variation	Too big; too small	✓	(✓)	(✓)	(✓)	✓	✓	✓	✓	(✓)			✓	✓	(✓)	✓
Extra material	Residual powder; closed pores	✓	(✓)	(✓)	(✓)	✓		(✓)	(✓)	(✓)			✓	✓		✓
Deformation	Warping; overall distortion	✓		(✓)		✓	✓	✓	✓							
Structure Defect	Broken/ missing features	✓		✓	✓	✓		(✓)	(✓)	(✓)						✓
Surface Defect	Uneven surface / waviness	✓					✓	✓	✓	✓	✓	✓				
	Surface texture; roughness	(✓)					(✓)			✓	✓	✓				
	Staircase effect	✓					✓			✓	✓	✓				
	Point defects (where support was removed)	✓					✓			✓	✓	✓				
Bulk Defect	Porosity	(✓)											✓	✓		
	Cavities	✓	(✓)	✓	✓	✓							✓	✓		(✓)
	Delamination	✓		✓	✓	✓										✓
Measurement Facts																
System	Possible **R**esolution	R	R (red)	R (red)	R (red)		R (green)	R	R	R (green)	R (green)	R (green)				
Sample	Geometry (**l**attice/ **d**ense/ **a**ny)	a	d	d	d	a	d	a (grey)	a (grey)	d	d	a	a	a	l	a
	Material (**m**etal/ **p**olymer/ **c**eramic/ **a**ny)	a	c/p	a	a	a	a	a (grey)	a (grey)	a	a	a	a	a	a	a
Efficiency	Costs	€ (red)	€ (red)	€	€	€	€ (red)	€	€	€	€	€	€ (green)	€ (green)	€ (green)	€ (green)
	Time	⏱	⏱ (red)	⏱	⏱	⏱ (green)	⏱	⏱	⏱ (green)	⏱ (green)	⏱	⏱	⏱	⏱	⏱ (green)	⏱ (green)
Defect detection	**L**ocal or **G**lobal	L	L	L	L	G	L	L	L	L	L	L	G	G	G	G

Legend:

✓	Possible	⏱ (green)	Fast (< 2h)	€ (green)	Cheap (< 100 €/h)	R (green)	Very high resolution(nm range)
(✓)	Possible with limitations	⏱	Medium (2h – 4h)	€	Medium (100 – 200 €/h)	R	Good resolution (µm range)
		⏱ (red)	Slow (> 4h)	€ (red)	Expensive (> 200 €/h)	R (red)	Low resolution (mm range)

6.2 Parts with Intended Defects

To evaluate the suitability of different measurement methods to identify typical defects occurring in additively manufactured lattice parts, the Wurtzit type structure as described in chapter 3 has been printed with LCM technology. The same structure was modified, four kinds of defects were intentionally introduced, printed as well and investigated.

*The findings have been published in Open Ceramics. For further information please refer to the following article: https://doi.org/10.1016/j.oceram.2020.100020 (Wilbig, J., et al., Defect detection in additively manufactured lattices. Open Ceramics, 2020. **3**.)*

6.3 Protocols for Defect Detection

The parts with and without defects presented before were investigated by weighing and volume determination using Archimedes method. Measurement protocols and results are presented in the following sections 6.3.1 and 6.3.2. Also, resonant acoustic testing was performed on parts with defects as described in section 6.3.3.

6.3.1 Protocol I: Defect Detection by Weighing

*The findings have been published in Open Ceramics. For further information please refer to the following article: https://doi.org/10.1016/j.oceram.2020.100020 (Wilbig, J., et al., Defect detection in additively manufactured lattices. Open Ceramics, 2020. **3**.)*

6.3.2 Protocol II: Defect Detection by Archimedes Method

*The findings have been published in Open Ceramics. For further information please refer to the following article: https://doi.org/10.1016/j.oceram.2020.100020 (Wilbig, J., et al., Defect detection in additively manufactured lattices. Open Ceramics, 2020. **3**.)*

6.3.3 Protocol III: Defect Detection by Resonant Acoustic Method (RAM)

Resonant acoustic testing is a volumetric global method, comparative to reference components, which consists of measuring the component's mechanical resonances in air and to compare them to the mechanical resonances of reference components defined as "good parts". A shift in frequency will be observed in the mechanical resonances of a "bad part". The method enables to detect the component's structural characteristics: flaws, process variations in manufacturing of the component etc. Anything that will cause a shift to the mass, stiffness, and damping of a component can cause a shift in the resonant frequencies. The method was not applied to the lattice Wurtzit type geometries presented before, but on spine implants made of titanium alloy as presented in Figure 41 and on lattice specimens made of CoCr with different amounts of intended missing struts shown in Figure 42.

Figure 41: Spine implant produced by Medicrea made of Titanium alloy

Figure 42: Lattice specimen produced by LNE/ NIST with intended missing struts made of CoCr alloy

Resonant acoustic testing is a very fast and simple method, which allows to detect defects in samples of any shape, size and material. It is a pass/fail test, which requires reference parts and do not allow to localize a defect. The method is easy to integrate in manufacturing process chains and dedicated to serial production. Typical deviations occurring in additively manufactured parts such as geometrical variations (distortion, warping, shrinkage) or internal defects as voids, pores, cracks or missing features are detectable.

Measurement system

The system used for testing was the Semi-Auto Non-Destructive Test System from The Modal Shop, MTS Systems Corporation as presented in Figure 43 with an impact hammer, a microphone, a digital controller and a software as main components.

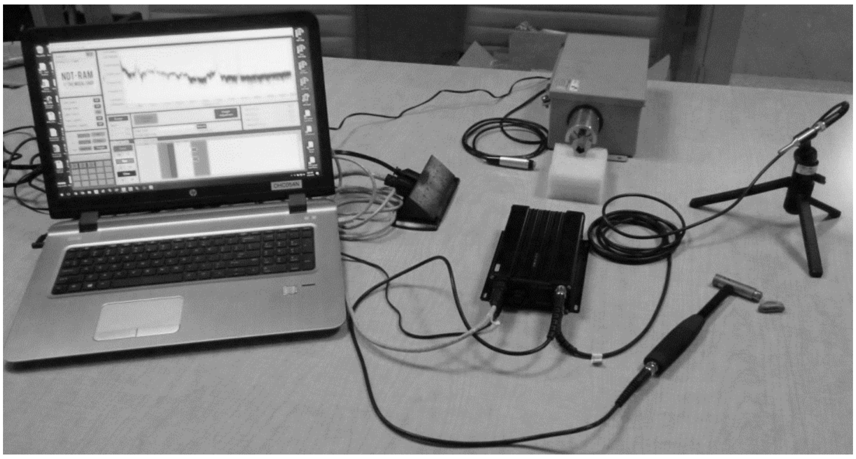


Figure 43: Semi-Auto Non-Destructive Quality Test System, The modal shop MTS Systems Corporation

Measurement protocol

The excitation of the part, to make it vibrate, is done by an impact hammer and the resulting acoustic frequency data is obtained with a microphone. The output of the microphone is converted into a frequency spectrum using a high-speed analog to digital converter performing a fast Fourier transform to determine resonant peaks. Finally, a statistical analysis conducted with software reveals if the test parts fall within the defined criteria or not.
Inspection step by step:

1. Impact of the tested part with a hammer tip;
2. Measurement, by a microphone (+Fourier transform), of the component's mechanical resonances: frequency spectrum;
3. Selection of 3 to 6 well defined frequency peaks in the spectrum of the reference parts that will be used as comparison criteria;
4. Comparison of the spectrum of the test parts with the criteria: all resonant peaks of the test part that fall outside the criteria are considered as bad parts/all resonant peaks of the test part that fall between the limit in frequency and amplitude of the criteria are considered as good parts;
5. Separation of good and bad parts.

Results

Applying RAM method on the parts with defects allowed to detect all visible defects in the spine implants and missing struts in the lattice specimen, as long as there were at least two missing struts compared to the reference part.

7 Recommendations for Prevention of Deviations

AM technology specific defects and possible measurement methods to detect them have been presented in previous chapters. This chapter describes in more detail possible ways of defect prevention for the Binder Jetting process and the Lithography-based Ceramic Manufacturing for each step of the process chain according to the diagram presented in Figure 44.

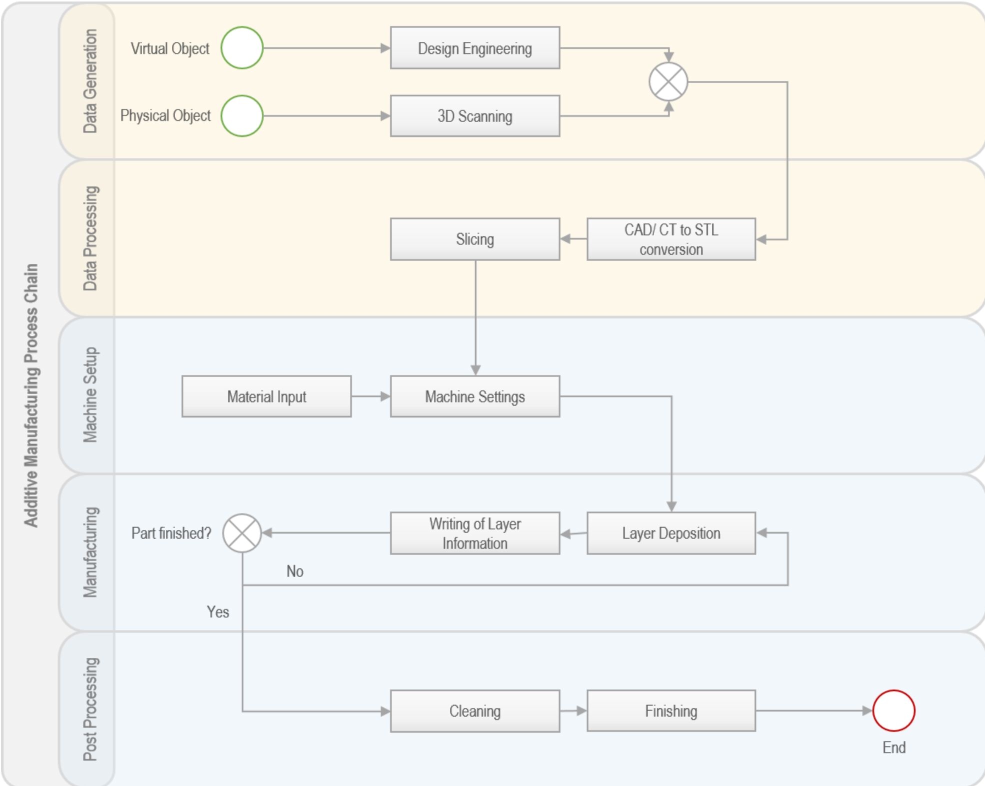


Figure 44: General Additive Manufacturing Process Chain

7.1 Binder Jetting

In the powder-based Binder Jetting process the quality of the fabricated parts depends mainly on the powder bed quality, on the precision and repeatability of the print head as well as on the interaction between powder and binding liquid and finally on the heat treatment during post processing. For ceramic parts, the post processing by heat treatment is most critical and prone to introduce defects.

<u>Data Generation</u>

- *Design*

The design should generally consider the volumetric resolution of the specific AM system used. The size of the designed parts should not be smaller than the minimum possible feature size of the system to avoid broken or disconnected or even missing features in the final part, like designed pores or channels which will be filled with material in the final part because they were designed smaller than the possible feature size. Design of support structures can help to provide a stable basis for the part, as the first layers tend to move during recoating. But after a few layers have been applied, the part is supported by the surrounding powder bed and no special support needs to be designed for overhangs etc. The expected factor of shrinkage due to thermal treatment should be considered already during design.

Data Processing

- *CAD/ CT to STL Conversion*

Virtual data obtained need to be converted into a machine-readable data format and separated into a sequence of two dimensional slices with specific thicknesses. While the surface properties of the part can be influenced by the resolution of the STL file, further errors can occur during the conversion as incorrect aligned triangles resulting in gaps in the surface. For complex geometries it is difficult to detect these gaps by visual inspection of the virtual part. Software tools are available to check the virtual parts for misaligned triangles and to repair them like e.g. MAGICS (Materialise, Leuven, Belgium).

- *Slicing*

The virtual model is separated in a certain amount of two dimensional slices with a specific thickness. The chosen thickness leads to a stair case effect, which is becoming more prominent with the slice thickness increasing. Powder properties as particle size should be considered when choosing the layer thickness.

Machine Set Up

- *Material Input*

Powder: The powder used for printing has direct influence on the final part quality. The particle size and particle size distribution define the surface roughness of the final part and influences the materials sintering behavior, the final part porosity and thus mechanical stability. Finer particles typically lead to lower surface roughness, better sinterability of ceramic parts and a higher achievable part resolution. Smaller particles allow smaller layer thicknesses and therefore a reduced staircase effect. Additionally, the part contours can be more precise. Too fine particles on the other hand show typically a poor flowability due to higher interparticle forces, tend to agglomerate and are harder to handle in the layer deposition process. They even tend to stick to the recoating system and, thus, can cause defects in the powder layer and in the powder bed.

A suitable particle size for Binder Jetting of ceramic powders is between 45 and 100 µm. Within this particle size range a homogeneous powder layer can be deposited. A mixture with smaller particle can increase the packing density of the powder, as spaces between bigger particles can be filled with the smaller ones. The powder bed density basically stipulates the density of the green part in ceramics printing.

Printing with particles smaller than 45 µm would be beneficial in terms of geometrical accuracy and sinter behavior but complicates due to agglomerations the deposition process. Equipping the printer with special accessories, like a gas flow assistance system can help to distribute even fine powder homogenously. For gas flow assisted powder deposition [17] the build plate is replaced by a porous plate (pores smaller than powder particles) to which a vacuum pump is connected. The pump creates a gas flow through the powder bed, compacting particles and thereby leading to higher powder bed density and green part density.

To avoid defects in the deposited layers and the printed part it is mandatory to know the powder properties and to consider them when choosing the printing parameter. Prior to printing the powder should be characterized regarding particle size distribution, flowability, densitiy (powder bulk and tap density) and chemical composition. The operator should be aware, that powder properties can alter during storage, they change when powder is re-used and they can also vary between different batches from one supplier.

Binding Liquid. The type of binding liquid is different for polymer and ceramic powders. When printing polymer particles a solvent is typically used to bind the single particles. It partially dissolves the surface of the polymers by chemical reaction and creates a connection between the single particles. Afterwards the solvent simply evaporates. Post processing with heat treatment is not necessarily required.

For printing with ceramic particles, the binding liquid is typically an organic binder which glues the single particles together and which is burned out during post processing. Especially for the organic binder it is important to check for the chemical composition and the boiling points of the different components to determine the appropriate temperature schedule for debinding.

Generally, the chosen binding liquid and the print head should be compatible. To achieve homogeneous parts, it should be ensured that the print head is working properly and missing jets are identified. Fully or partly clogged nozzles lead to no or redirected droplets, creating areas within a layer, that are not properly covered with binding liquid. This can have an influence on the mechanical properties of the final part and its shape. To avoid this, the print head should be checked prior to each print job. This can be done by printing test patterns on e.g. paper. This way, defective nozzles become visible. Some machines also have a camera system installed, which allows to watch the droplets coming out of the single nozzles. This facilitates not only the identification of defective nozzles but also the evaluation of droplet formation and the adjustment of print head settings.

When defective nozzles have been identified, the print head can be purged to clean clogged nozzles or the non-working nozzles can be disabled in the software and are replaced by working nozzles during printing. To avoid clogged nozzles in the first place, the system should ideally be constantly in use. If it is not used, the print head should be covered to avoid drying of the binding liquid in the print head. The print head should be covered as well when it comes to generation of powder dust during refill of material, as the fine powder particles can react with residual liquid at the print head and promote clogging.

Additionally, the performance of the print head is influenced by the environment. Temperature changes can lead to variations in the binder viscosity and surface tension and thus in droplet formation. Measuring the weight of the single droplets is a simple way to detect changes and to consider them when setting the printing parameter.

- *Machine Settings*

The most important parameter to be adjusted for printing is the desired saturation, i.e. the desired amount of binder for one volumetric element, the voxel. When the saturation is chosen too high, too much binder is applied to the powder bed, resulting in loss of part accuracy as the binder diffuses beyond the desired contour. It can also lead to total damage of the part, when binder accumulates on top of the powder and comes in contact with the recoater that applies the next layer. If the amount of binder is too low on the other hand, the printed part will lose mechanical stability. It is recommended to choose the highest possible saturation, that leads to the desired geometry contour.

The saturation depends on the parameter shown in the following equation [18]:

$$S = \frac{V_b}{\left(1 - \frac{PR}{100\%}\right) * dx * dy * LT}$$

V_b is the volume of a single droplet applied by one nozzle of a print head [pL]. *PR* represents the powder packing rate, which is calculated by the ratio of the powder bulk density (the freely settled untapped density) to the theoretical density of the material. The lateral area on the powder bed for one droplet is defined by $dx * dy$, which represents the spaces between two droplets in x and y direction [µm]. These spaces are related to print head velocity, spitting frequency and print head offset. LT represents the layer thickness [µm], which typically should be chosen higher than the maximum particle size. Otherwise defects in the powder bed can be created during recoating.

In summary, the saturation provides the ratio of binder applied to the free volume in a voxel, not filled with material, considering all important printing parameter. Thus, it is an applicable parameter to evaluate the amount of binding liquid used to connect a certain amount of powder particles in a voxel. It is not uncommon that the saturation exceeds the value 1, as the binding liquid evaporates or dries when it is applied, and a higher amount of binding liquid can be ejected than free space is available in the powder. Which saturation is most suitable for the powder and part geometry chosen should be determined by doing test prints.

Any recoating settings like spread speed or vibrations should be set according to visual inspection. They should lead to a smooth powder bed without ripples, groves or pits.

Additive Manufacturing

When the AM machine is prepared, printing starts with layer deposition. Subsequently, the layer information is printed on the applied layer by local deposition of binding liquid in the desired shape of the part's cross section.

- *Layer Deposition*

The powder layer is typically deposited with a roller or doctor blade and should result in a smooth layer without any defects. Any defect present in the powder bed can influence the final part quality. If the layer is not smooth, the recoater should be checked. There could be grooves on it or more likely residual material which agglomerates on the roller or on the blade. As already described, the powder flowability is an important factor for deposition as well. Agglomerations can also be a result of humidity in ambient environment. Generally, the environmental conditions should be constant for a reliable printing process. Defects can also result from recoating settings, which are not optimal. Velocity can be adjusted, or the powder feed ratio changed, so that enough powder is applied to cover the entire build plate.

- *Writing of Layer Information*

During preparation of the machine a test pattern was printed, and the proper function of the nozzles checked. Despite of this it is possible, that clogging occurs during printing. This depends on the binding liquid used. Especially when using binding liquid to glue ceramic particles, clogging of the print head is likely. This can be avoided by additional cleaning cycles during printing, where the print head is purged. Depending on the software used it is also possible to have an offset between the position of the nozzles for every new layer. This way the loss of binding liquid resulting from a not properly working nozzles can be compensated in the following layer. When the binding liquid is printed, a short drying time for each layer can improve the green part stability. The ProMetal Type RX-D 2005 printer used for printing ceramic parts for example comes with a heating station for curing the binder every layer.

Post Processing

Post processing methods vary for polymer and ceramic parts. The polymer parts are left in the powder bed for some hours to let them dry and to let the solvent evaporate. Afterwards the parts are cleaned with compressed air or brushes from residual loose powder. A mild temperature treatment can support solvent evaporation. Then, the parts are ready to use or can be infiltrated to improve mechanical strength.
For ceramic parts, the green body needs to be dry before removing it from the powder bed. The green part is very fragile and needs to be cleaned carefully with compressed air and brushes to remove loose powder. The final heat treatment at high temperatures allows the burnout of the binder and the sintering of the ceramic particles. In the following, the single steps of drying, cleaning and heat treatment are described more precise.

- *Drying*

Drying of polymer parts was achieved by leaving them in the powder bed over night to give the solvent time to evaporate and to reduce the possibility of introducing defects during handling and cleaning, as the part is stronger in the dry state.
Drying of ceramic parts occurs already during printing, as every layer is dried under the installed heating unit. This improves the mechanical stability of the green parts and reduces also the possibility of breaking or deforming the part during cleaning.

- *Cleaning*

Both polymer and ceramic parts are cleaned from loose powder using compressed air and brushes. For the scaffolds printed, cleaning was not always successful. While the polymer powder was hard to remove from the cells inside the scaffold, even when the geometry was scaled up, the powder in the cells of the ceramic parts could be removed very easily just by slightly tapping the parts. Probably the evaporating solvent reacts with the powder trapped in the cells and impedes cleaning. The design of lattice structures need to take material properties into account. Designed pore sizes should be adjusted accordingly.

Handling of the parts during cleaning can lead to deformation of the contour, to missing material due to breaking of fragile areas and when any support structures need to be removed, also to surface defects which are hard to avoid.

- *Finishing*

Printed polymer parts have been dried at 40°C in an oven for several hours after cleaning to help residual solvent to faster evaporate. Ceramic parts in contrast, underwent a high temperature treatment to achieve fusion of the single particles. The heat treatment comprises of a debinding and a sintering step.

For debinding, the furnace is set to a temperature at which components of the binder are becoming volatile. During this critical process step, defects are easily introduced to the part, as gas is formed which has to leave the part. If heating occurs too rapidly, too much gas is generated at once and cracks or blisters can be formed, impeding the mechanical properties of the part. To avoid any deformation, the whole process should be kept slow for ensuring a homogeneous temperature over the entire part. A certain dwell time is required at the debinding temperature, while the time depends on the amount of binder in the part and on the part geometry. Debinding of thinner parts like lattice scaffolds is much faster than debinding of dense parts. The dense ceramic cubes printed and presented in chapter 4.1 showed significant deformation, which could be reduced by using a slower heating rate (60 K/h instead 300 K/h) and a longer dwell time (2h instead of 1h). Thus, gas is generated more slowly and has more time to escape from the parts body without creating pressure inside and forming cracks. Mechanisms of debinding are still subject of research. [19]

After binder burnout, the printed ceramic part is sintered. Sintering in general is the process of densification of powder-based green parts, leading to a solid body with a residual porosity depending on the green part properties. Densification and solidification happens due to grain growth and elimination of pores under high temperature. Governed by the reduction of surface energy, the shape of the particles changes due to material transport mechanisms as diffusion. Overall shrinkage occurs while the shape of the part is kept. [20]

Sintering processes can be based on solid state or on liquid phase sintering. Advantage of liquid phase sintering is the presence of an additional component in the powder, which has a lower melting point than the main component, forming a liquid phase at the sinter temperature which fills the space between the solid particles due to capillary forces. A higher density and improved mechanical properties can be achieved this way. [20]

The sinter temperature depends on the chosen material and powder properties like particle size. As a reference point, for oxidic materials $T_{sintering}$ is typically between $0.7*T_{melting}$ and $0.8*T_{melting}$. [20]

To avoid any introduction of defects during sintering, the appropriate temperature, dwell times and heating rates for the specific material and part geometry should be defined with the aid of existing literature or by performing sinter studies. The phenomenon of shrinkage is well known for ceramic parts and a homogeneous sintering critically depends on a homogeneous density of the printed powder compact. To produce a part in a desired geometry and size, the factor of shrinkage should be defined and considered already during design.

If sintering is not intense, high residual porosity and low mechanical stability will be the result as the connection between the single particles is weak. Is sintering too intense on the other hand, residual porosity decreases indeed but grain growth is enhanced significantly, and sporadic pore growth can occur, which in turn impedes the mechanical stability of the part. In addition, the mechanical stability can be reduced in case of material phase changes at the temperatures used for sintering. This for example happens for tricalcium phosphate. At temperatures around 1200°C a phase transition from β- to α-TCP occurs, associated with transformation of the crystal structure, which leads to different densities and the formation of cracks [21]. To avoid defects, material phase diagrams should be considered.

For a better control of the temperature it is also recommended to measure and record the temperature inside the furnace. A uniform heat distribution inside is mandatory to avoid anisotropic shrinkage.

7.2 Lithography-based Ceramic Manufacturing

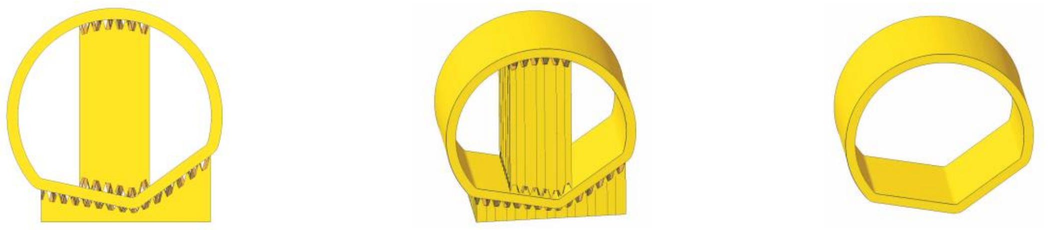
Critical to the quality of parts printed with LCM technology is the composition of the suspension, in which ceramic particles need to be homogeneously dispersed as well as the temperature treatment of the printed parts, which is necessary for debinding and sintering of the ceramic particles. Recommendations for prevention of defects along the process chain are described in the following.

<u>Data Generation</u>

- *Design*

During lithographic printing of ceramic suspensions green parts are formed – this means the ceramic powder is embedded in an organic matrix. To receive dense ceramic components, the binder has to be removed and the parts have to be sintered. This process relies on the elimination of the porosity which is created by the removal of the organic binder and hence, is accompanied by shrinkage. Therefore, it is necessary to scale the printed part in order to end up with the desired dimensional accuracy in the sintered state.

Regarding the desired building orientation, support structures may be necessary. If there is no connection between the currently cured layer to the last layer, this connection must be formed by support structures. Also overhang structures may need support in order to stabilize the structure during the printing process and prevent deformation of the part. However, the part should initially be orientated so that a big area presents the link to the building platform, and following layers become continually smaller. This especially is helpful with increasing geometry height, because of the increasing tilting forces. Therefore, a good connection to the building platform is crucial to prevent peeling, as the parts are built upside-down and could fall into the vat.



Design of support structure *LCM-process* *Removing of support structure*

Figure 45: Beginning from the design of a support structure to the final component without support

Even if additive manufacturing offers new possibilities in design, there are anyway some guidelines to take into account. As the processed material is ceramic, analog to conventional shaping technologies sharp edges and notches should be avoided, because of the appearance of stress peaks and subsequently flaws or cracks at such points. Therefore, corners and edges should ideally be rounded. Furthermore, also large differences in wall thickness could be difficult to fabricate. The sintering process for thicker parts takes longer than for thinner ones, which leads to differences in shrinkage and hence shrinkage strains, again resulting in defects.

wrong correct

Figure 46: Avoiding of sharp edges and corners, left – wrong design, right – correct design.

If the desired geometries are designed with respect to the printing guidelines, printing can be facilitated and optimal results can be expected.

Data Processing

- *CAD/ CT to STL Conversion*

STL-files can directly get loaded into the Cerafab-software of Lithoz GmbH. In the software it is possible to multiply or delete parts, as well as rotate, and position them at one's option at the virtual building platform. Once you are confident with your designed job, and all the machine parameters (see below) are set, it is possible to have a look at the preview, which shows all single layers of the job, to check if nothing is damaged. Also, the estimated building time and material use are calculated. By sending the job to the printer, the actual slicing-process automatically takes place.

Machine Set Up

- *Material Input*

Concerning processability, there are several requirements a photocurable ceramic suspension (slurry) has to fulfill in order to be printable.
The photoreactivity of the slurry is crucial for the proceassability in the LCM-technology. The photoreactivity is characterised by the light curing depth – which means how far the light is transmitted into the slurry so that photopolymerisation and thus, solidification, occurs. The light curing depth has to be higher than the thickness of the individual layers of the component, to ensure adhesion between the layers.
Also, the slurry-viscosity affects the processability in the LCM-technology. The lower the viscosity, the easier the coating of a homogeneous slurry-film in the vat can be realized.
The solids content is another crucial aspect to enable good material properties in the final part. An increase of the solids content could lead to an improvement of the mechanical properties of the sintered ceramic like the bending strength or relative density. Also, the shrinkage due to densification processes during sintering is decreased, which leads to a decrease in shrinkage strains. However, an increase of the solid content goes along with an increase in viscosity and a decrease in light curing depth. Therefore, the solid content has to be selected wisely for an optimal outcome.
To receive homogeneous green parts and subsequent homogeneously densified sintered components without flaws or defects, also the quality of the used slurry has to be assured. The ceramic powder has to be suspended uniformly within the organic binder system and agglomerates have to get eliminated. Furthermore, the particle size distribution of the ceramic powder has a significant impact on the processability of the slurry. The largest particles have to be smaller than the chosen layer thickness of the given print job. Otherwise the vat or the printed parts get damaged.
Lastly, the slurry has to be stable – at least for the duration of one whole printing process, ideally significantly longer. If the slurry separates in its components while printing due to physical instability, inhomogeneous parts are formed. If the slurry experiences chemical degradation processes like gelation, the printing results are also affected.

- *Machine Settings*

The quality and precision of the fabricated parts is also dependent on the applied processing parameters during the lithographic shaping of the parts. The LCM-process provides the opportunity of defining numerous parameters to enable the processability of different slurries that vary either in the composition of the photocurable matrix or in the chemical nature or properties of the inorganic particles. Variations of the material will have consequences on the photoreactivity and the rheological properties of the ceramic suspension and thus influence the processability. Crucial parameters for the shaping process include the layer-thickness (thickness of each individually cured layer), the used light intensity as well as the exposure time per layer. By changing the exposure time, the applied energy-dose can be varied. The used energy dose and the layer thickness should be adapted, to guarantee interlayer-bonding. If the layer thickness is bigger than the curing depth at applied energy-doses, there will not be proper adhesion between the layers and printing is not possible. Therefore, the light curing depth of the slurry at certain energies has to be known to design the right printing parameters.
Another aspect is the rotation speed of the vat, which determines the homogeneous slurry coating in the vat. Because the slurries are too viscous to flow by itself into the gaps, which were left from the last

layer, the vat is rotated to recoat the slurry. Therefore, the rotation speed compromises printing time, but is necessary for a homogeneous slurry coating to enable the curing of homogeneous layers.
The slurry is solidified where the light hits the material, so that the current layer is built between the last layer and the vat surface. To peel the cured layer off the vat surface, the vat is able to perform tilt-movements. The tilting-speed is a variable machine setting, which should be adapted to the stiffness of the emerging green body. The more flexible the parts are, the lower the tilting-speed should be chosen to avoid tearing.
Another parameter, which could be adjusted, is the settling time – the waiting time before light exposure. It depends on the size of the area, which has to be cured. The lager the area, the longer should be the settling times to allow the slurry to flow in the space between the last layer and the vat. Therefore, the settling time also depends on the viscosity of the slurry. With lower viscosities lower settling-times are possible due to quicker flow behavior.

Additive Manufacturing

- *Layer Deposition*

A thin slurry layer is deposited by the rotation of the vat and a fixed blade (typically 100 – 300 µm). However, the height of the deposited slurry layer does not represent the layer thickness with which the structure is built. The layer thickness is defined by the gap between the building platform and the vat. It is adjusted by the movement along the z-axis of the building platform. After every layer, the building platform is moved upwards, so that the vat can be rotated to recoat the slurry. After that, the building platform is lowered again dipping into the deposited slurry layer until the desired layer thickness is reached (typically between 10 and 100 µm).

- *Writing of Layer Information*

The selective curing of each layer is done by digital light processing (DLP). Using this approach, the whole layer information is transferred simultaneously via a digital micromirror device (DMD). As this methodology relies on the use of a pixel array, the original layer geometry is approximated by an algorithm. Based on the result of this algorithm the respective mirrors of the DMD are tilted and reflect light onto the photocurable ceramic suspension and trigger photopolymerization which leads to selective solidification in exactly these areas.

Post Processing

After the shaping process, green bodies are formed – the ceramic powder is embedded in an organic matrix. Due to the viscosity of the slurry, excess material can remain adhered to the surface of the printed component. Therefore, the parts have to be cleaned after printing.

- *Removing from the building platform*

When the print job is finished, the building platform together with the printed parts can be removed from the printer. Depending on the used slurry and the printed geometries, the adhesion of the parts to the building platform varies. The printed parts can be removed from the platform by cutting them off with the use of scalpels, saws or by breaking them from the support structures (if those have been used).

- *Cleaning*

The manufactured green bodies have to get cleaned due to excessive slurry adhered to the compound-surface. The cleaning can be done while the parts are still placed on the building platform, or after their removal by the use of compressed air and solvent. However, it is recommended to use an amount of solvent not more than necessary, due to the possibility of infiltration of the part and subsequently resulting in cracks during debinding and sintering. After the cleaning-process, the parts should be dried by the use of compressed air or also paper-towels. As the green bodies are sensitive to air humidity, desiccators or drying cabinets are recommended for longer storage.

- *Finishing*

The last critical step to obtain dense ceramic parts is the thermal post-processing. This involves debinding, where the organic matrix is removed by treating the parts at elevated temperatures. This step is usually the most time-consuming, due to the sensitivity of flaw-formation. During thermal debinding, the organic matrix is pyrolyzed and decomposed. If the decomposed matrix is removed too quickly, a pressure is formed within the component. This pressure leads to the formation of cracks. To avoid this, tailored heating rates and dwell times are used to ensure homogeneous heating throughout the whole component.

After debinding, the obtained parts (white parts) are then sintered in a high-temperature furnace to give the dense ceramic bodies. Given the nature of the ceramic material, debinding and sintering can also be conducted in the same furnace if the necessary sintering temperature is not too high.

8 Summary

Typical geometrical deviations identified in parts printed within the frame of MetAMMI using different AM technologies have been presented in this report along with recommendations for their detection and prevention. Parts produced by Binder Jetting, laser-based Powder-bed Fusion and Material Extrusion have been investigated as well as parts manufactured using Vat Photopolymerization and Lithography-based Ceramic Manufacturing. Liquid, solid and powdery feedstocks were processed to fabricate metal polymer and ceramic parts with dense and lattice geometries. For lattice parts, outer and overall geometrical deviations as well as porosity and strut thickness have been quantified and compared.
An analytical toolbox has been developed and measurement protocols based on inspections on parts with intentionally introduced typical defects. Methods for defect detection by simple weighing, by volume determination using Archimedes method and by resonant acoustic testing have been demonstrated.
Weighing and volume determination by Archimedes allowed to detect deviations leading to variations in mass and volume of around 20%. Finally, measures for prevention of defects in Binder Jetting and Lithography-based Ceramic Manufacturing process chain have been described comprehensively.

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