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Materials and measurements for additive manufactured customized human implants

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Abstract. Designing and manufacturing medical implants are complex tasks. These implants have to fit numerous requirements in their geometries and materials for a proper behavior. To determine the exact composition of their materials is important, and by the use of GDOES we can get reliable and quick results about that. This presentation demonstrates the results of a GDOES measurement taking the implants' quality requirements into account.

1. Introduction

Designing and manufacturing medical implants are complex tasks. In most cases we have to create customized, irregular geometric geometries made of different expensive materials which have to fulfill various requirements to be able to behave naturally in vivo. Taking these into account one can easily tell that the traditional manufacturing of these implants could cost a lot of money and time. These obstacles, however, can be eliminated by additive manufacturing technologies. This presentation demonstrates the Selective Laser Melting (SLM) procedure, which uses metal powder to build parts, reviews the most critical factors effecting the results of the SLM, the made geometries. It also mentions the important requirements regarding the contact surfaces for in vitro application. Materialistic adequacy is a key component. To quickly and accurately determine the material composition the use of „Glow-discharge optical emission spectroscopy (GDOES)” can offer a proper solution.

2. Selective laser melting

The Selective Laser Melting (SLM) is an additive manufacturing technology. The term „additive manufacturing” includes the layer-by-layer building technologies, which were mostly used in the Rapid Prototyping (RP), but today, by the fast development of these technologies we have become able to manufacture medical implants fulfilling most of the quality requirements.

SLM uses metal powder (Ti6Al4V, 316L stainless steel, etc.) and laser beam to build layers. The laser beam's absorbed energy melts the top of the powder bed in a preset layer thickness by scanning it in the cross sections of the built part. The melted pool's dimensions have to secure a total melted connection between the connecting layers and between the adjacent scanned lines so a perfect cohesion connection can be created [1]. The mechanism of action of the scanning is shown on figure 1. After melting a layer, the powder bed is sunk by the amount of a layer thickness, then a mechanism (blade, roller, etc.) covers the top of the powder bed with new powder layer taken from a powder container, and the overflowing



powder is collected by a different powder container [2]. The basic schematics of this kind of equipment can be seen on figure 2. These steps continue until the part is totally manufactured.

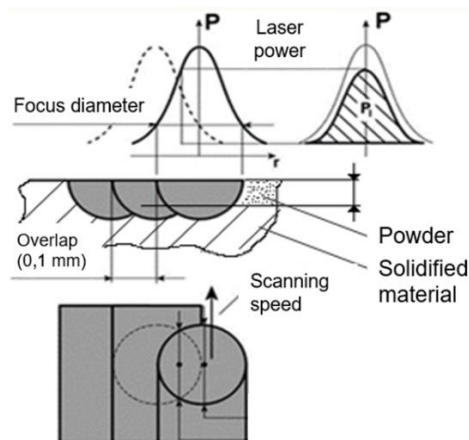


Figure 1. The mechanism of SLM [1]

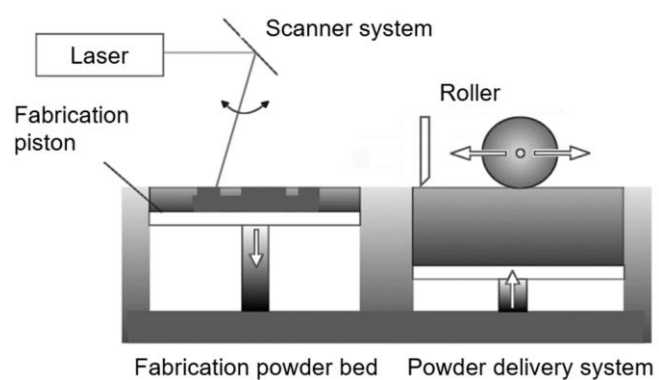


Figure 2. Schematics of an SLM equipment [2]

3. Designing and manufacturing implants

3.1. The bone

The bone contains organic (39%) materials (95% collagen, 5% proteoglykan), inorganic (49%) materials (calcium hydroxiapatite crystals) and fluids (12%). Bone has two types: cortical (solid) and cancellous (spongy) bone. The cortical bone is hard, rigid, this wears most of the stress, but it has low breaking elongation (~2%). The cancellous bone is more porous, can maintain a higher elastic deformation (~75%) but can not bear high loads. The proportion of these two types, age, sex, way of life (e.g. sportsman or not), the function and placement of the bone (craniofacial or tibia) form the bones' mechanical attributes, so it is hard to exactly determine that [3]. However, it can be declared, that bone and implant metals represent a way different mechanical behaviour, as shown in Table 1.

Table 1. Mechanical properties of implant metals and bone [4]

Material	E modulus [GPa]	Yield strength [MPa]	Ultimate strength [GPa]
316L SS	190	221-1213	586-1351
Co-Cr alloys	210-253	448-1606	655-1896
Titan	110	485	760
Ti6Al4V	116	896-1034	965-1103
Cortical bone	15-30	30-70	70-150

3.2. Implants

Implants are medical devices made of biologically satisfying materials, built into the human body by purpose and are partially or totally covered by tissue. In the designing phase it is important to pay attention to the effects when operating or maintaining the implants, it must not ransom invasive reactions in the organism [5].

The requirements of implants [6, 7] are biocompatibility (the organism should not reject it), bioactivity (cells can stick to and grow on its surfaces), good surface quality, appropriate porosity, pore shape and size (proliferation can start and tissue can grow) and proper mechanical properties (so it will not damage the ambient bone tissues). Latter is defined by the Wolff's law, which describes the stress shielding, where stress is worn by the more rigid implant instead of the more flexible bone (figure 3). This can lead to osteoporosis in the ambient area of the implant, that can elad to the losing of the built in part.

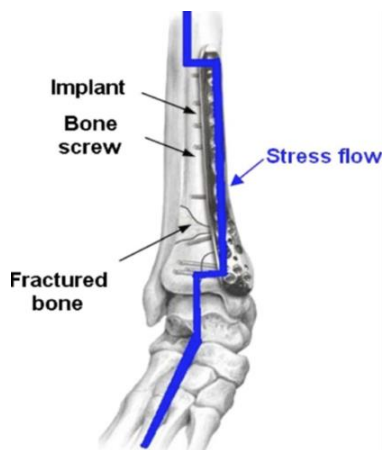


Figure 3. Bone to implant stress shielding [8]

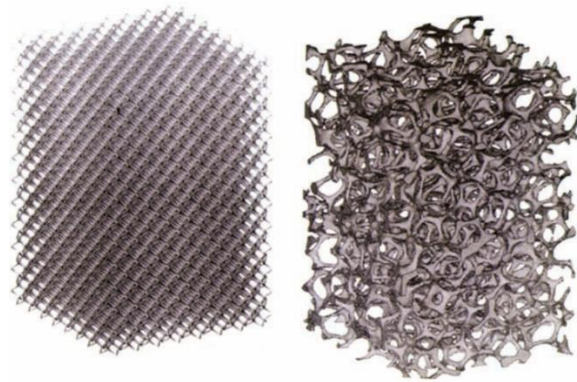


Figure 4. Open cellular structures for implants [9]

4. Main factors effecting the SLM-built parts' quality

The results of the SLM are dependent on many parameters of the technology, these can be categorized into 4 groups [10]:

- attributes of the laser beam (wave lenght, beam diameter ont he surface, energy density etc.)
- material attributes (powder size, melting point, flow-rate etc.)
- scanning parameters (scanning speed, hatch distance, ratio of overlapping, scanning strategy etc.)
- ambient factors (temperature, atmosphere, residual O₂ level etc.)

However, it is impossible to vary all paramteres in most cases. The usually variable and most important parameters are described in the followings.

4.1. Energy density

It has the biggest impact on the manufacturing process, and it's formula is:

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

where E: energy density [J/mm³], P: power of laser beam [W], v: scanning speed [mm/s], h: hatch distance [mm], t: layer thickness [mm] [11]. Energy density means the energy implied into a unit of space. In general it can be stated that by the right adjustment of these parameters – and all in all the energy density - any material can be manufactured to the required quality [12]. An example of the effect of energy density can be seen on figure 5 through the porosity of the created parts.

4.2. Scanning strategies

Scanning strategy (figure 6) means the way the powder layer is scanned. It greatly affects the developing heat profiles, textures and grain growth direction during the solidification. Choosing the strategy influences the manufacturing time and the product mechanical (residual stresses and surface quality). For a long time, the uni-direction (x or y) scanning was used (figure 6 (a)), which caused different heat

gradients, poor quality and high surface roughness. Using alternating directions while scanning can lead to better results (figure 6 (b)). The best quality can be reached if the whole cross section is divided into smaller areas (e.g. 5x5 mm squares), and these are scanned individually using the zig-zag strategy rotated by 90° after each other (figure 6 (c)), then the strategy applied to the layers are rotated by 45-90°.

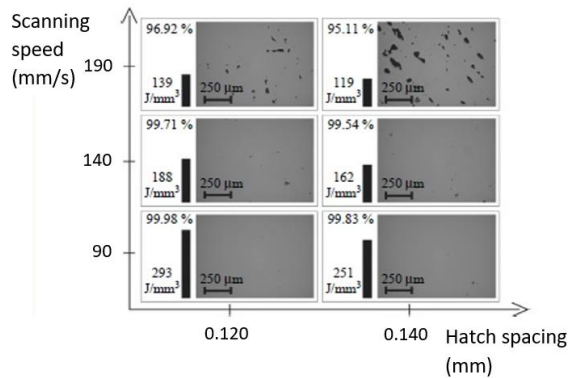


Figure 5. Effect of energy density on porosity [11]

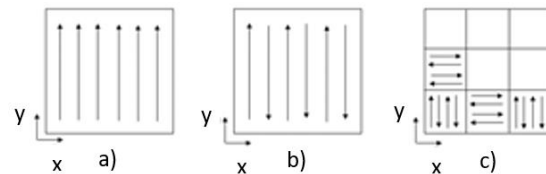


Figure 6. Scanning strategies. (a) uni-directional, (b) alternating directional (zig-zag), (c) chesstable-like

5. Material composition measurements

Implants have strict biological and healthcare requirements, so their materials have to maintain a composition fulfilling these. Regarding the laser technology any kind of disturbance during the manufacturing process can change the proper composition. For example, ambient oxygen or nitrogen may diffuse into the part, or overheating of the powder can cause a partial vaporization of a component in the alloy despite of the well-balanced process control of the machine.

This is the reason, why it is necessary to use a fast and accurate material analysing measurement to quickly check the process quality through inspecting samples made in the same process along with prototype samples, and can analyse the presence of polluting elements, gas molecules (H, O, N) and carbon besides of the main alloy elements.

5.1. GDOES optical emission spectroscopy

The Glow Discharge Optical Emission Spectroscopy, GDOES is capable of carrying through quality and quantity analysis of metallic and non-metallic materials. The Glow Discharge Source is a Grimm type discharge tube characterized by a special arrangement of the electrodes: The two electrodes of the DC Current Source are set up of a cylindrical hollow anode and a cathode. The sample is placed directly onto the cathode and hence, is switched as cathode itself. The sample must seal the anode tightly so a vacuum can be generated (figure 7). Thus, a flat and preferably smooth sample surface is required.

The sample is placed into the Glow Discharge Source and switched as cathode. The Glow Discharge Source is filled with argon gas under low pressure. Direct Voltage is applied between a hollow anode and the cathode ($\hat{=}$ sample). Due to the energy input of the DC voltage the argon atoms are ionized resulting in the formation of a plasma. Argon cations are accelerated towards the negative sample surface and knock out some sample atoms (“sputtering process”). The knocked out sample atoms diffuse into the plasma where they collide with high-energy electrons. During these collisions energy is transferred to the sample atoms promoting them to excited energy states. Returning to the ground state the atoms emit light with a characteristic wavelength spectrum (figure 8). In the spectrometer the light is dispersed into its spectral components, which are registered by the detection system. The intensity of the lines is proportional to the concentration of the corresponding element in the plasma.

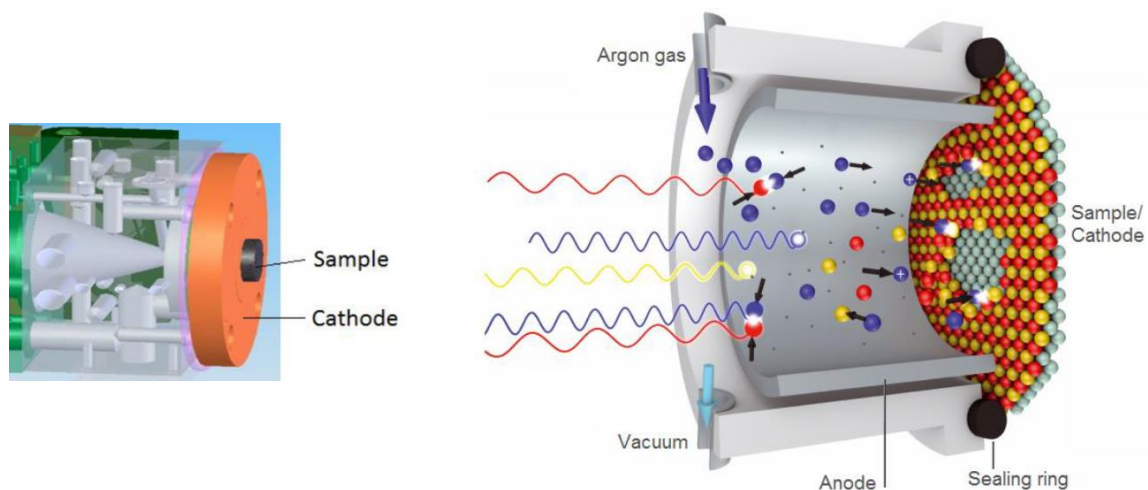


Figure 7. Schematic presentation of the Glow Discharge Source. The sample seals the anode tightly. [13]

Figure 8. Sputtering process in the Glow Discharge Source. Yellow/red spheres: sample atoms; blue spheres: argon atoms or cations [13]

5.2. Results

We used GDOES to analyse an SLM-made sample regarding its depth-dependant composition. The graph containing the results can be observed on Table 2. and figure 10, where the lines with different colours represent different elements (in weight %) in 1-10-100-1000x magnification. The sample is a Ti6Al4V part made by SLM, which can be seen on figure 9 along with the analysed spots. It was stored in an open air environment for a long time ($>> 72$ hours) before the measurement.

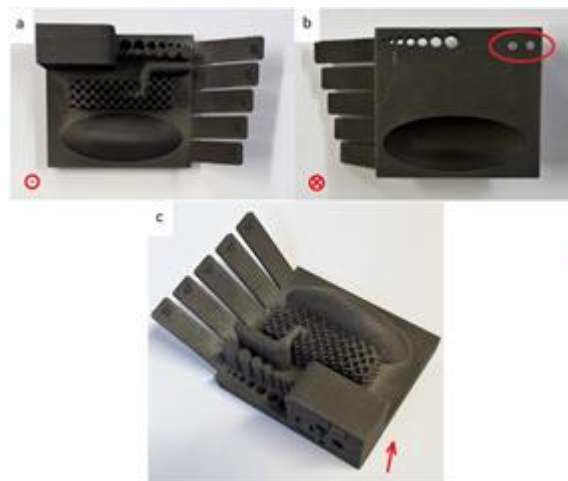


Figure 9. The sample. (a) top view, (b) bottom view with the analysis spots, (c) axonometric view. The z direction of the building is shown on every picture

The deviation of the composition (figure 10.) is stopped after 4-5 μm depth (refer to Table 2, column 1). This is where the equilibrium composition of the elements are stable. It can be observed, that in the top layers (0-4 μm) the oxygen and nitrogen are present in a way higher ratio than they should have been according to the theoretical composition. This may be caused by the storing method of the sample.

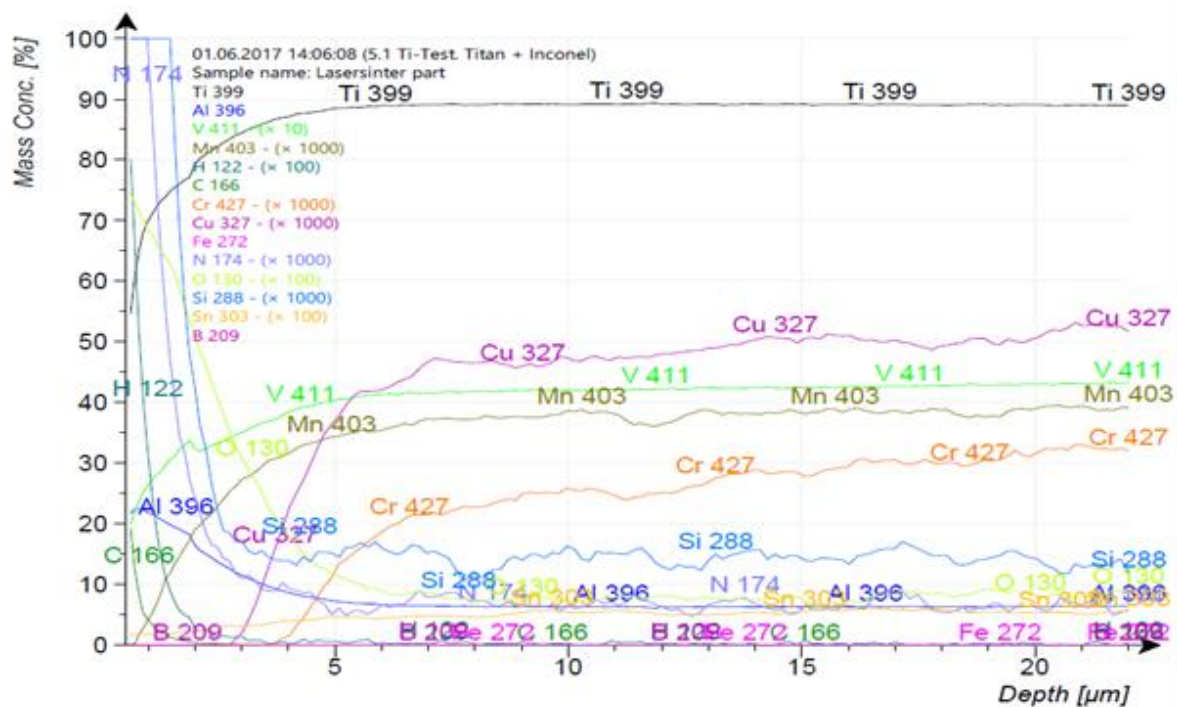


Figure 10. GDOES results of the material composition according to sampling depth [13]

Table 2. Material composition of specified elements in different depths

Depth, μm	Ti %	Al %	V %	C %	H %	N %	O %
2,6119	71,746	19,263	2,849	4,956	0,231	0,072	0,637
4,7782	85,942	9,972	3,692	0,064	0,008	0,01	0,216
6,9616	88,764	6,85	4,105	0,023	0,005	0,006	0,091
9,1781	89,15	6,404	4,179	0,018	0,003	0,007	0,075

The structural differences can also be seen by Table 2 at the depth of 2,6119 μm . The amount of Ti is almost 20% lower than the equilibrium level ($\approx 90\%$), while Al represents a 19,263% quantity instead of 5,5-6,5% and vanadium is present with a 2,849% amount instead of 3,5-4,5%. This ratio of Al and Ti can tell, that a non-equilibrium structure or alloy is present in this depth that can be the cause of different events, e.g. the rapid heat abstraction occurring during the building phase. These effects should be investigated with further targeted examinations, especially because if aluminium contacts or diffuses into bone, it delays, slows or prevents the growth of bone cells and tissue. Aluminium salts are also capable of causing chondrogenesis and osteogenesis [14]. The non-equilibrium ratio of Al can mean that most of it is not stored safely in a compound phase, so as an implants surface this composition may be easily diffuse into the ambient organs and damage them.

In deeper layers (deeper than 5 μm) the elements' ratio stabilizes to a certain level. Comparing this to EOS's standardized material composition regarding this powder material, it can be seen that after 9 μm all elements are the specified tolerance.

6. Summary

As we could see, designing and manufacturing customized implants is a task relying on many factors and different scientific areas. The quality of SLM made metal human implants depends on many parameters which can be factors of the technology, storage, handling, structural design, the patient herself and so on. The material measurement results showed us key effects – e.g. patient-health-risking changes - occurring in the material composition during an SLM manufacturing cycle which could have

been unknown otherwise. We have to identify the parameters causing these effects, so the proper setting of these parameters can be the key to create implants that can ensure the better life quality of patients. That is why we are going to do further analysis with greater attention on these specific parameters among the rest of them.

7. Acknowledgement

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The researchers are grateful for the support.

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