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The Education, Scholarships, Apprenticeships and Youth Entrepreneurship

EUROPEAN NETWORK FOR 3D PRINTING OF BIOMIMETIC MECHATRONIC SYSTEMS

MODULE 8

Intelligent (Smart) Materials

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1 Introduction in Additive Manufacturing technologies

1.1. The principle of Additive Manufacturing technologies

Additive Prototyping Technologies (AM) differ fundamentally from material removal processing technologies (cutting, EDM, laser processing) and redistribution processing technologies material (casting, injection, forging, stamping) by the fact that the parts are obtained by adding layer by layer material using a CAD file.

These technologies have emerged grace a result of the achievements and advances made in the field of fine mechanics, numerical control, laser technology, computers, software, and the new materials development.

These new Additive Manufacturing technologies have started to grow in importance due to the efforts of manufacturers to reduce design times up to marketing, as well as the costs of assimilating and manufacturing new products. The specificity of these additive manufacturing processes is their ability to make parts and complex three-dimensional objects, starting from a CAD file, without the need for it use of machine tools or certain tools. The basic element of prototype additive manufacturing technologies is "the section". [1-86]

The pieces are quantified in sections and made using a repetitive process of construction, section by section, reducing a three-dimensional problem to one flat. This dimensional reduction leads to a decrease in accuracy and quality surfaces due to the scale effect.

The steps required for the additive manufacturing of a part are as follows:

-designing the three-dimensional (3D) model of the part, using a design program computer aided (CAD);

-transferring the CAD model to the sectioning processor. The best-known method of sectioning is the approximation of the model with flat triangular elements.

- sectioning the 3D virtual model with parallel planes to the working plane of the rapid manufacturing machine of prototypes and generation of orders for control equipment of the machine.

-the construction of the part (material, supports required during the model, how will be added a new layer, marking the contours for each section, marking the area between the exterior and interior contour of a section.

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- cleaning and finishing of the part (operations in which the supports used at construction and excess material are eliminated).

Additive Manufacturing Processes were initially used to produce prototypes, in the case of small, unique series production. Soon, however, these manufacturing processes Additive will directly produce functional parts with high precision and shorter manufacturing time, either metallic or other materials, successfully replacing classical manufacturing technologies that used expensive machine tools.

Regarding solid CAD modelling, Additive Manufacturing systems are becoming an important and motivating factor for companies that produce solid modelling systems, such as: Solidworks, Unigraphics, I-DEAS, Catia, Inventor, Onshape, AutoCAD, Pro / Engineer, etc.

Models obtained by Additive Manufacturing optimize the design of a new model or modernization of an existing one, these models allow the physical visualization of the product and improving communication between producer and beneficiary.

Testing a product manufactured using Additive Manufacturing technologies depends on three factors material, size, and design. The tests performed must lead to a visual acceptance, understanding of construction, product functionality and finalization sizing elements.

Additive manufacturing technologies have and will play an important role in many industrial fields, from the field of Machine Building to medicine, aerospace, architecture, thus giving technology a strategic importance for companies that use these technologies. [1-85]

1.2. Classification of additive manufacturing technologies according to the materials used

Additive prototyping technologies can be classified by materials used (figure 1.1), namely:
-technologies that use liquid polymers as a base material, and solidification is realizes by the light impact from a special light source or from a small laser power (stereolithography) or by heating (thermal polymerization);
-technologies based on melting, deposition and resolidification of the material. These allow the use of metals (DMD, LMD technologies) as well as plastics or liquids (FDM process, ballistic particle processing).

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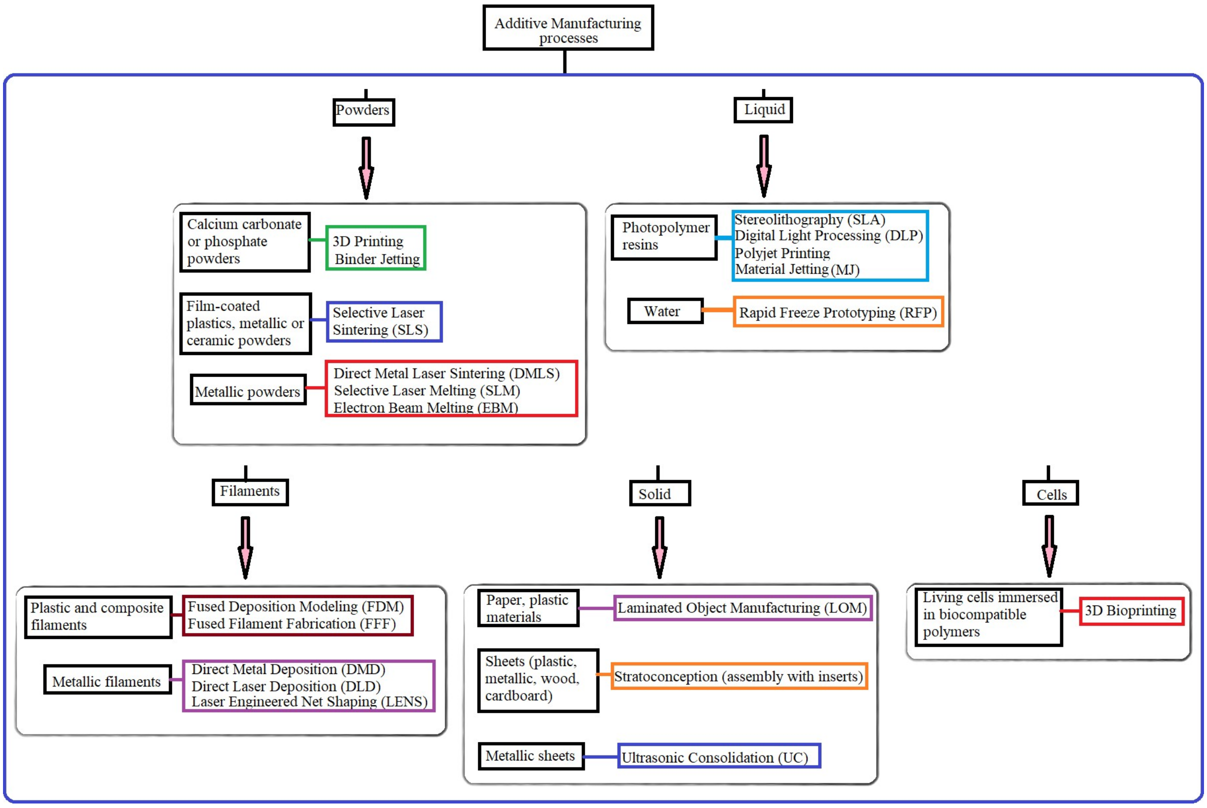

Figure 1. Classification of additive manufacturing technologies according to the materials used

-other processes use powder as raw material. [2] Binding of particles to powder can be made by melting in the contact area between particles of the same kind or with particles of another material which will be constituted as a special complementary material for this process (selective laser sintering SLS, DMLS, SLM), or gluing particles in areas of interest with a special substance (three-dimensional printing or gluing) – Binder Jetting.

-some processes use solid raw material, especially thin foils, as LOM or Stratoconception technologies.

Some processes are made by gluing or welding thick foils to produce the shape requested. Other processes use the semi-polymerization of plastic foils which are fixed together by another light curing. [1-86]

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2. Plastic materials used in Additive Manufacturing

ABS and PLA are the most common FDM (Fused Deposition Modeling) printed materials and are typically similar in cost. ABS has superior mechanical properties but is harder to print compared to PLA. PLA is ideal for 3D prints where aesthetics is important.

Table 1. The mechanical properties of Acrylonitrile Butadiene Styrene (ABS)

Properties	Values	Units
Density	1.0-1.4	g/cm3
Poisson's Ratio	0.35	-
Shear Modulus G	1,03-1,07	GPa
Melting Temperature	200	ºC
Glass transition temperature	105	ºC
Thermal Conductivity	0,25	W/m-K
Extruded Temperature	200-230	ºC
Heat Deflection Temperature, 1,81 MPa	81	ºC
Young's modulus	1,79-3,2	GPa
Tensile Strength	29,8-43	MPa
Compressive Strength	76-78	MPa
Elongation at Break	10-50	%
Flexural modulus	2,1-7,6	GPa
Hardness Shore D	100	
Izod Impact Strength	58	kJ/m2
Yield Strength	28-120	MPa
Standard Tolerance	+/-0.05	mm
Biodegradable	-	-
Melt flow	12-23	g/10min
Rockwell Hardness	R102-R104	

Due to its lower printing temperature is easier to print with and therefore better suited for parts with fine details. ABS is best suited for applications where strength, ductility, machinability and thermal stability are required. ABS is more prone to warping. The mechanical properties of ABS are presented in Table 1. [1-86]

Other materials used frequently in FDM technology are the filaments of Polyethylene Terephthalate PET $(C_{10}H_8O_4)_n$ and Polyethylene Terephthalate Glycol (PETG). The mechanical properties of PET material are presented in the Table 2 and the comparison concerning mechanical properties between the common materials used in FDM technology, PLA, ABS and

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HIPS is shown in the Table 4. In table 3 are shown the mechanical properties of PLA for 3D printed material.

Table 2. The mechanical properties of Polyethylene Terephthalate PET $(C_{10}H_8O_4)_n$

No	Mechanical and chemical properties	U.M.	Value (unit)	Obs.
1	Density	g/cm^3	1.455 – cristalin 1.37 - amorphous	1.38 – at 20ºC
2	Tensile Strength	N/mm^2	74-cristalin 55-amorphous	-
3	Compressive Strength	N/mm^2	125	-
4	Flexural strength	N/mm^2	90	-
5	Torsion strength	N/mm^2	-	-
6	Shear strength	N/mm^2	-	-
7	Elongation at break	%	50-cristalin 150-300 - amorphous	-
8	Ball penetration hardness	Kg/m^3	1370	-
9	Rockwell Hardness	-	R100-cristalin R90-amorphous	-
10	Charpy shock resistant (uncracked)	kJ/m^2	3.6	-
11	Charpy shock resistant (cracked)	kJ/m^2	2.5	-
12	Melting temperature	ºC	260	-
13	Glass transition temperature	ºC	67-81	-
14	Notch test	kJ/m^2	3.6	-
15	Vicat Temperature(VST)	ºC	82	-
16	Extruded temperature	ºC	220-250	-
17	Liniar expansion coefficient	-	7	$(*10^{-5}K^{-1})$
18	Specific Heat	cal/gºC	0.28	$(JK^{-1}*kg^{-1})$
19	Thermal conductivity	W/mK	0.15-0.24	-
20	Boiling point	ºC	350	-
21	Volume resistivity	Ω*cm	$4*10^{16}$ – cristalin $2*10^{16}$ - amorphous	-
22	Surface resistivity	Ω	10^{13}	-
23	Water absorption (ASTM)	%	0.5-0.6 – cristalin 0.6-0.7 -amorphous	/24h
24	Viscosity	cP	75000-90000	Low-viscosity PET at high-viscosity PET
25	Dielectric rigidity	kV/mm	16	-
26	Melt flow	g/10min	35,08	230ºC
27	Young's Modulus (*E*)	MPa	2800-3100	-
28	IZOD Impact strength	J/m2	140	-

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Table 3. The mechanical properties of PLA (Polylactic Acid)

Properties	Values	Units
Density	1.25	g/cm3
Poisson's Ratio	0.36	-
Shear Modulus G	2.4	GPa
Melting Temperature	173	°C
Glass transition temperature	60	°C
Thermal Conductivity	0.13	W/m-K
Extruded Temperature	160-220	°C
Heat Resistance	110	°C
Young's modulus	3.5	GPa
Tensile Strength	61.5	MPa
Compressive Strength	93.8	MPa
Elongation at Break	6	%
Flexural strength	88.8	MPa
Hardness Shore D	85	A
Impact Strength	30.8	kJ/m2
Yield Strength	60	MPa
Standard Tolerance	+/-0.05	mm
Biodegradable	yes	-

Table 4. Comparison concerning mechanical properties between the common materials used in FDM technology, PLA, ABS and HIPS

Polymers	HIPS			ABS			PLA		
	OV	SD	SE$\bar{x}$	OV	SD	SE$\bar{x}$	OV	SD	SE$\bar{x}$
MFI (g/10 min)	7.5 ± 0.20	0.16	0.11	8.76 ± 0.16	0.13	0.09	13.52 ± 0.11	0.09	0.06
Young's modulus (MPa)	112.5 ± 0.12	0.09	0.06	175 ± 0.11	0.09	0.06	47.9 ± 0.10	0.08	0.05
Yield stress (MPa)	3.44 ± 0.21	0.17	0.12	0.49 ± 0.21	0.17	0.12	0.27 ± 0.16	0.13	0.09
Glass transition temp (°C)	100.41 ± 0.16	0.13	0.09	109.76 ± 0.2	0.16	0.11	62.57 ± 0.21	0.17	0.12
Peak load (N)	80.8 ± 0.11	0.08	0.06	207 ± 0.2	0.16	0.11	282.4 ± 0.20	0.16	0.11
Peak strength (MPa)	4.21 ± 0.16	0.13	0.09	10.78 ± 0.11	0.09	0.06	14.71 ± 0.16	0.13	0.09
Peak elongation (mm)	1.9 ± 0.20	0.16	0.11	4.75 ± 0.16	0.13	0.09	5.13 ± 0.16	0.13	0.09
Percentage elongation at peak (%)	3.0 ± 0.11	0.09	0.06	6.0 ± 0.15	0.12	0.08	7.0 ± 0.10	0.08	0.05

3. Photopolymerizable resins used in Additive Manufacturing

In the SLA (Stereolithography) and DLP (Digital Light Processing) technologies are used photocurable vinyl- or epoxy- functional oligomers for photopolymerization. In figure 2 are presented the transparent resins used in dental domain, manufactured by SLA. The principle

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of manufacturing for both technologies is presented in figure 3. In table 5 are presented the mechanical properties of Bisphenol A Ethoxylate Diacrylate resin. Other resins used in SLA manufacturing are the polyurethane resins. In figure 4 can remark the SEM image for the resin sample. [1-86]

Figure 2. Transparent resins used in dental domain manufactured by SLA

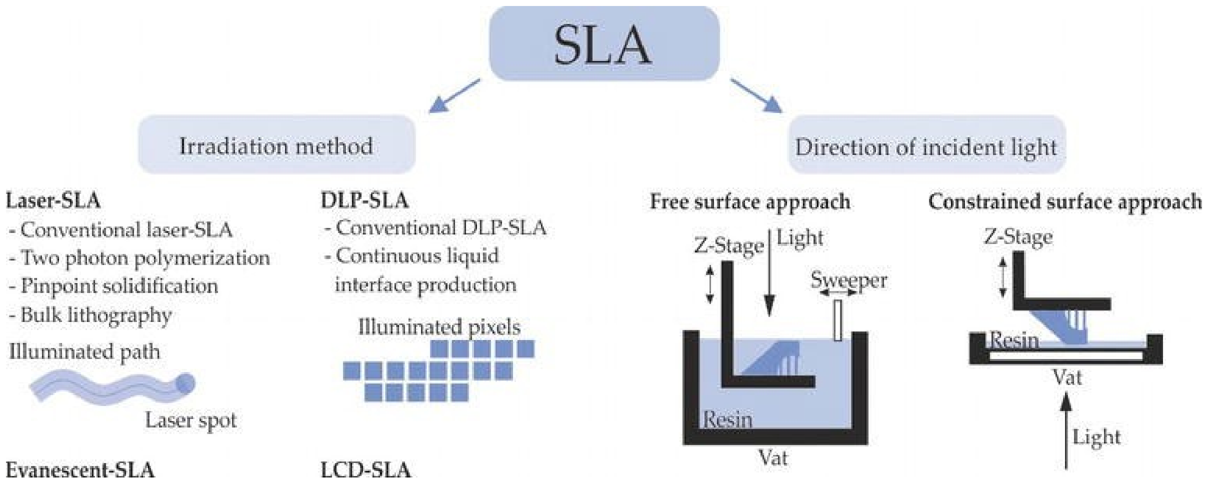

Figure 3. The principle of manufacturing for both technologies: SLA and DLP

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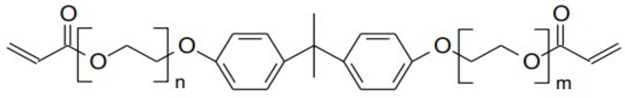
Table 5. The mechanical properties of Bisphenol A Ethoxylate Diacrylate

Bisphenol A Ethoxylate Diacrylate



n + m ~ 4

INTRODUCTION

EBECRYL 150 is an ethoxylated bisphenol A diacrylate commonly used as reactive diluent in UV/EB cure applications. EBECRYL 150 can improve the cure response, hardness, and chemical resistance of UV/EB curable coatings and inks while maintaining good adhesion, and without imparting brittleness.

PERFORMANCE HIGHLIGHTS

EBECRYL 150 is characterized by:

- High reactivity
- Moderate viscosity
- High refractive index

UV/EB curable formulated products containing EBECRYL 150 are characterized by:

- Hardness
- Chemical resistance
- Good adhesion
- Improved wetting

The actual properties of UV/EB cured products also depend on the selection of other formulation components such as oligomers, additives and photoinitiators.

SPECIFICATIONS[1]	VALUE
Acid value, mg KOH/g, max.	5
Appearance	Clear liquid
Color, Gardner scale, max.	2
Viscosity, 25°C, cP/mPa·s	1150-1650

TYPICAL PHYSICAL PROPERTIES	
Density, g/ml at 25°C	1.14
Flash point, Setaflash, °C	>100
Functionality, theoretical	2
Refractive index (n_D at 20°C)	1.5294
Vapor pressure, mm Hg at 20°C	<0.01

TYPICAL CURED PROPERTIES[2]	
Tensile strength, psi (MPa)	6300 (43)
Elongation at break, %	9
Young's modulus, psi (MPa)	180000 (1241)
Glass transition temperature, °C[3]	41

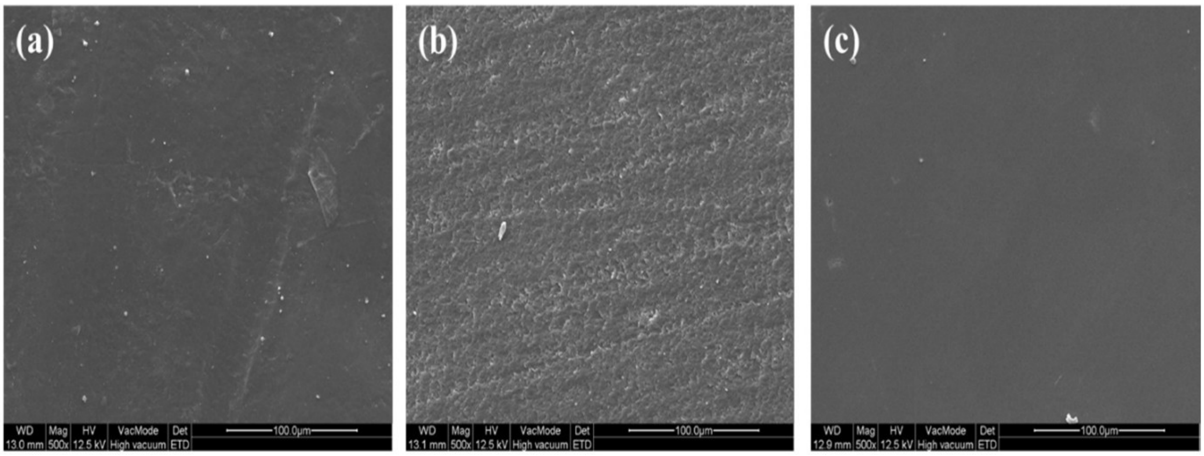


Figure 4. FE-SEM images (x500) of an unpolished resin sample as – SLA fabricated (a) after sanding (b), and (c) after sanding and Chemical Mechanical Polishing

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4. *Metallic powders used in Additive Laser Manufacturing (ALM)*

These technologies have emerged grace a result of achievements and progress made in the field of fine mechanics, numerical control, laser technology, computer technology, computer software and new materials.

Laser additive manufacturing is a set of processes that allow layer by layer fabrication by adding material, creating a 3D physical model starting from a numerical model.

Direct laser additive fabrication of metal powders is performed by DMLS, SLM, EBM, DMD, LM, LENS, DLF processes and use pure powders metallic, obtaining parts that can be functional immediately, not being necessary always a post-sintering treatment. [1-86]

Classification of the main technologies of Laser Additive Manufacturing, in according to the manufacturing principle is as follows:

I. Melting the metal powder bed

SLM - Selective Laser Melting

LBM - Laser Beam Melting

DMLS - Direct Metal Laser Sintering

EBM - Electron Beam Melting

II. Melting of conditioned metal powders with binder (polymer)

SLS - Selective Laser Sintering

SLA – Stereolithography

LM – Layered Manufacturing

FDM - Fused Deposition Modeling

3D Printing

III. Metal powder design and laser melting

DMD - Direct Metal Deposition

LENS - Laser Engineering Net Shaping

DLF- Direct Light Fabrication

3D Laser Cladding

LFF - Laser Freeform Fabrication

Laser Consolidation

EasyClad

LDT - Laser Deposition Technology

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IV. Wire technologies

Wire Deposition

EBF3 - Electron Beam Freeform Fabrication

DMD - Direct Metal Deposition (NTiC)

WAAM - Wire and Arc Additive Manufacturing

Indirect additive manufacturing of metal powders is carried out by SLS process, because the metal powder is conditioned with a binder for making the green piece, after which a post-sintering treatment is required to improve mechanical properties.

During post-sintering treatment performs depolymerization, burning of the binder used in the first phase of sintering with laser. For this reason, the SLS process is an indirect process of additive manufacturing of metal powders.

Each ALM process has its advantages and disadvantages concerning the residual stresses in the material after sintering / melting.

It is noted that by processing SLM, DMLS, EBM by melting powder bed, obtaining parts with very fine details with a very high complexity degree, but the speed of the process is low and the parts present mechanical properties quite low compared to the parts obtained by DMD with wire. [1-86]

The DMD (Direct Metal Deposition) process with metal wire is similar in principle FDM (Fused Deposition Modeling) procedure.

SLM, DMLS and EBM technologies are used for parts with fine details, with a high degree of complexity of shapes, but the manufacturing speed of the processes is slow. The mechanical properties are lower than in the case of DMD technology. Use fine-grained powder between 5-30 microns.

LMD technology (Laser Metal Deposition) is used for manufacturing medium complexity parts that present better mechanical properties, than SLM, or DMLS technologies. This technology uses granular powder larger of the order between 50-150 microns.

DMD (Direct Metal Deposition) is used for the manufacture of semi-finished products with simple geometric shapes.

The mechanical properties are good, like the forged parts.

The DMD wire process is mainly used for repair metal parts and has a high manufacturing speed, very good mechanical properties of the parts, but it cannot make complex shapes and very fine details.

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DMD manufacturing systems are similar as CNC centers, having 5 axes and allow10 depositions of different metal materials on different surfaces of metal parts by performing the movements due to the movements allowed on the 5 axes.

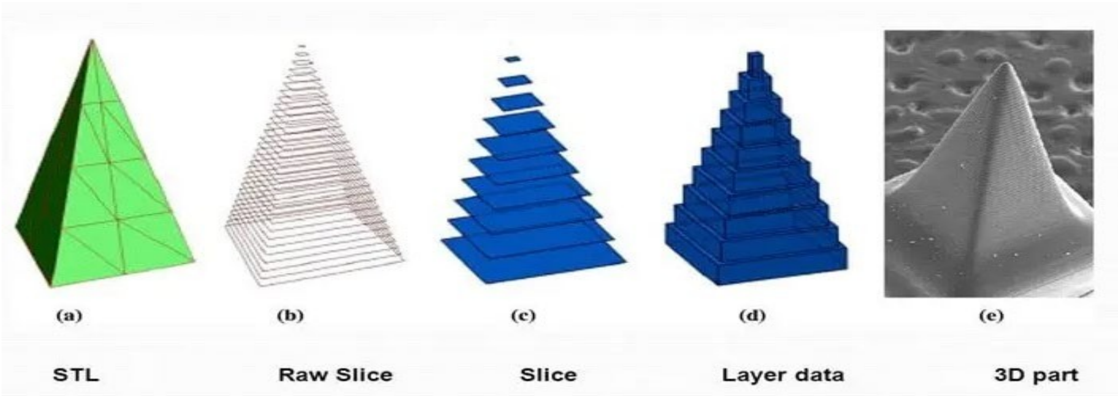

Figure 5. The general processing principle of Additive Laser Manufacturing technologies

The general processing principle of Additive Laser Manufacturing technologies (ALM) is presented in Fig.5.

Laser Additive Manufacturing of Metal Powder (ALM) must hold account for the next parameters:

1-temperature profile.

2-thermal gradient of metal powder.

3-speed solidification.

4-speed cooling.

For large series production, manufacturing systems are dotted with 1kW power lasers.

ALM manufacturing systems use different sources concerning the 3D model, namely:

-conversion CAD files (* STEP, * IGES);

-3D scan (optical, RX,…) of the existing model.

-3D models designed and exported from various design software (Blender, Catia, Solid Works, ProEngineer,…), saved as * stl or * gcode files.

Main uses of additive laser manufacturing (ALM) technologies are shown in figure 6.

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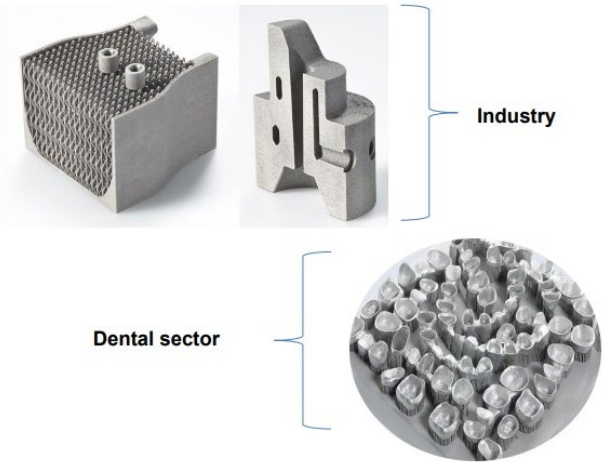

Figure 6. The main uses of additive laser manufacturing technologies (ALM): in industry and in medical domain

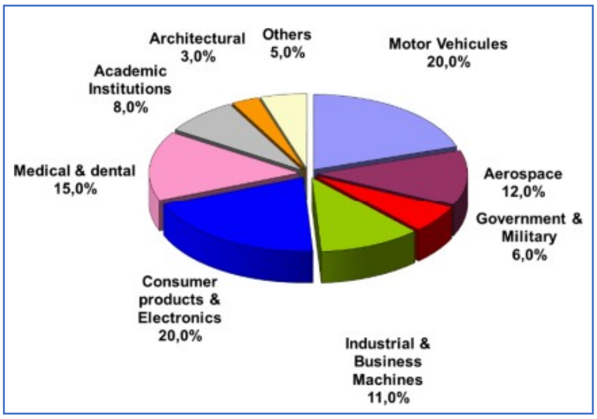

Figure 7. Applications domain of Additive Laser Manufacturing

The technological breakthrough brought by additive manufacturing in the field of manufacturing, by making it possible to rethink the design and production engineering of systems, pushes the industrial sector to increase the share of investment in this sector promising to manufacture. The evolution of the turnover of additive manufacturing to 6/36 Metal additive manufacturing: technologies and opportunities the global scale will be multiplied by 4.9 to reach the sum of $10,800 million by 2020, as in figure 7. [1-86]

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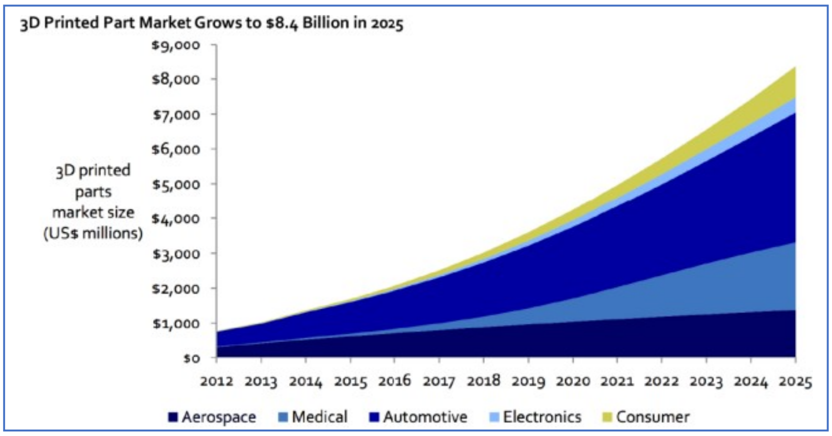

Figure 8. Market development, by industrial field, of additive manufacturing and its evolution

Among the major industrial fields interested in this new technology: automotive, aerospace and to a lesser extent, biomedical and wind power, as in figure 8.

SLS technology using stainless steel powder is a process of indirect sintering, as the powder is conditioned with a binder to lighten the formation of bridges between grains, and the part obtained by SLS sintering, cannot be used immediately, because it does not have the necessary mechanical strength. For this reason, green piece, as it is called, must necessarily endure another fasting treatment sintering in the oven for 30 minutes at 1100°-1200°C, to be realized final sintering between the metal grains of the powder and to increase mechanical strength of the piece. Selective laser sintering uses a high-power laser, generally up to 25W. The laser selectively sinters the powder by scanning cross sections generated from a 3D model, data from a CAD file or from a 3D scan. After each cross-section is sintered, a new layer of powder is applied with the help of a roller, and the process is repeated until the piece is completed. The systems of SLS manufacturing usually use a pulsed laser because the density of the part depends mostly on the laser power and less on the sintering time. SLS systems preheat the powder in the powder bed just below the point of melting, to facilitate laser sintering. Parts manufactured by SLS technology do not require the production of supports to be sintered, because the bed of unsintered powder surrounding the part can be used as a support. This process allows the creation of parts with geometries complex, which are difficult to achieve through traditional technologies. [1-86]

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A noticeable design aspect is the fact that it is impossible to manufacture an element that is hollow inside, but completely closed. This is due to the fact that it is impossible to drain the unsintered powder inside the element completely closed.

SLS manufacturing systems have started to become affordable in the market, but there are still obstacles in terms of high-power consumption up to 5kW and because of the temperatures that must be controlled with a precision of 2°C, for the three preheating, melting and storage steps, before removing the part.

This process is like direct metal sintering technology DMLS laser and SLM selective laser melting technology, the three technologies using the same concept but differing in technical details. For SLS technology, the powder is conditioned with epoxy resin binder, and

laser power and sintering temperature is lower than in the case DMLS and SLM technologies. In the case of DMLS manufacturing the metal grains of the powder are not covered with binder, pure metal powder is used which is sintered, and the parts can be used immediately after sintering, without the need for further post treatment sintering.

The metallic powders used in DMLS and SLM processes are obtained by different methods, as: HDH (Hydrate-Dehydrate process), gas atomization, plasma atomization and plasma rotating electrode process.

Selective Laser Melting (Selective Melting Laser) SLM is a technology relatively new and differs from SLS technology because the metal powder is melted due to the high power of the laser beam above 25 W, obtaining a density of the material up to 80-90%, almost identical to the density obtained in the case casting, but they differ in the crystalline structure obtained, as well as the mechanical properties or the porosity obtained.

The SLM process allows parts to be built to form close cumulatively the net shape components. Some SLM machines use the powder with a single component, such as pure Ti alloy powder. Powders are usually produced by ball milling or atomization. [1-86]

The powder bed laser melting/sintering process does not only have advantages with regard to the geometry of the part. Indeed, the powder bed requires a surface plane to be spread by the roller, if the surface is not plane, the roller may hit the part and thus damaging the machine. Another important point is the fact that the laser powder bed fusion constructions most often exhibit anisotropy in the construction direction with an elongated grain along this axis. This phenomenon is due in part to the reflow of layer $n-1$ when layer n has been integrated. This "natural" orientation therefore leads to an anisotropy of the mechanical

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properties (figure 9). It is however possible to attenuate this phenomenon by applying treatments thermal post manufacturing (TTH) to homogenize the structure.

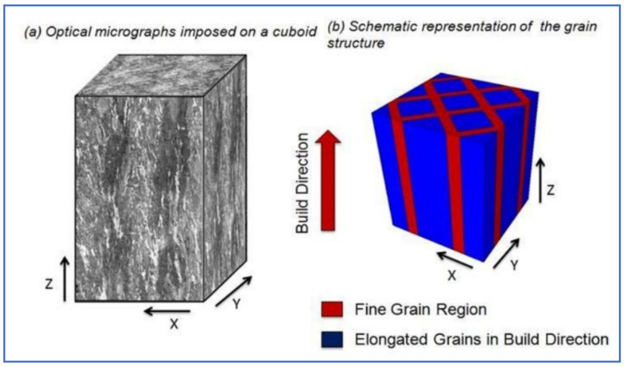

Figure 9. Anisotropy of the microstructure of parts built by SLM

Figure 10 presents additive manufacturing technologies performance concerning surface quality, part size, geometric complexity, manufacturing time, machine availability (number of machines available in the world), static properties, fatigue strength, fabrication cycle in industry, and, as can be noted, the DMLS process assures the best surface quality, geometric complexity, mechanical properties, and machine availability, but cannot be used to obtain large size parts and the manufacturing cycle is low. In comparison with other additive manufacturing technologies, such as EBM (electron beam melting), LMD (laser projection/laser metal deposition), DMD (direct metal deposition), only SLM (selective laser melting) and DMLS technologies can be used in dentistry.

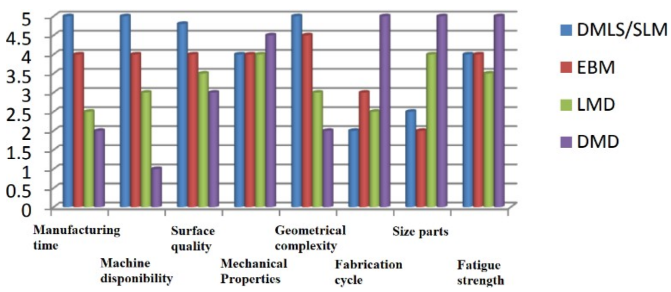

Figure 10. Comparison between the Additive Laser Technologies

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The most used technologies for the manufacturing metallic powders for DMLS are the hydride-dehydride process, water atomization, gas atomization, plasma atomization, electrode induction melting gas atomization (EIGA), plasma rotating electrode process (PREP), centrifugal atomization and plasma spheroidization.

The most common method of metal powder production for DMLS is plasma atomization. The elemental feedstock is melted under an air or inert gas blanket or under vacuum. The chamber is then backfilled with gas to force molten alloy to pass through a nozzle where high-velocity air, N, He, or Ar gas impinges the powder grains onto the flowing melt and breaks it up. The powder is mostly spherical, with some asymmetric particles and satellites present. A satellite is when a smaller particle sticks to a larger one during solidification. Grain size ranges from 0 to 500 microns. The most used powders that can be produced using this method are based on Ni, Co, Fe Ti, and Al alloys.

Characteristics of the powder particles at the end of the manufacturing process, using different standards,-are shown in Table 6.

Titanium cannot be directly brought in the powder state because it presents increased ductility compared to other crystallized metals, crystallinity being defined by the ratio of crystalline c/a over the theoretical value 1.633, which allows splitting grains after multiple plans with an atomic density close to the base or median plane.

Titanium is highly reactive to warm and oxide refractory materials so that the production of powders by mechanical means has the advantage of allowing the possibility to keep it at high purity level without being necessary to take special measures and expensive protection measures. The technology for obtaining titanium powder by hydride-milling-de-hydride systems is based on the behaviour of titanium to hydrogen. The solvus curve in the diagram is steep and provides the diffusion of a large amount of hydrogen in the solid solution. [1-86]

Upon heating under equilibrium conditions, for temperatures above 450°C, hydrogen forms a metastable hydride —TiH_2. This can be brought to ambient temperature in the atmosphere without decomposition or by a complex reaction. It is extremely brittle and may be easily sieved to give a particle size fraction distribution range. In the case of vacuum heating, the hydride decomposes, liberating hydrogen. The SEM analyses, using a JEOL JSM-5600, for Ti6Al4V are shown in Figure 11 and Figure 12.

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Table 6. Standards (ASTM, ISO, EN) for powder properties used in additive manufacturing [86]

AM Powder characteristics	Powder Type	Symbols	Techniques	ASMT Standard	ISO Standard	EN Standard
Size and shape	Metallic powders	Φ [μm]	SEM	B822	13322	-
Specific density	Metallic powders	$\rho_{specific}$ [g/cm^3]	Gas pycnometer	B293	12154	-
Apparent density	Non-free flowing metallic powders	ρ_{app} [g/cm^3]	Hall apparatus	B212	3923/1	3923
Apparent density	Non-free flowing metallic powders	ρ_{app} [g/cm^3]	Carney apparatus	B417	3923/1, 4490	4490
Apparent density	Metallic powders	ρ_{app} [g/cm^3]	Arnold meter	B703	-	-
Apparent density	Refractory metals and compounds	ρ_{app} [g/cm^3]	Scott volumeter	B329	3923/2	-
Tap density	Metallic powders	P_{tapped} [g/cm^3]	BT-1000	B527	3953	3953
Average particle size	Metallic powders	d_{60}	Fisher sub-sieve sizer	B330, C72	10070	-
Powder sieve analysis	Metallic powders	-	Sieve analysis equipment Westmoreland	B214	4497,2591	24497
Particle size distribution	Metallic powders and related compounds	d_{10}, d_{60}, d_{90}	Light scattering	B822	13320, 24370	-
Flowing rate	Free-flowing metallic powders	Flow time (s) for 50g	Hall apparatus	B213	4490	4490
Envelope specific surface	Powder bed under steady flow	S_v [m^2/g]	Measurement of air permeability	-	10070	196-6

The analyses emphasized two predominant forms of particles: blocks with flat facets—almost smooth—and sharp edges and peaks and foam particles, with a large number of microscopic cavities. This type of granulometry powder is not good for DMLS, because it influences the powder flowing rate and the sintering process quality.

The main mechanical characteristics of Ti6Al4V powder are: elastic limit 0.2% $Rp_{0,2}$= 815 MPa, elongation at break = 10%, Vickers hardness = 375 HV , elastic modulus = 229 GPa, mass density = 8,336 g/cm^3, corrosion resistance < 4 μg/cm^2, and thermal expansion coefficient = 14,5×10^{-6} K^{-1}.

The SINT-TECH company and ISO 9001 and ISO 13485 standards propose a range of powders suitable for the process developed by Phenix Systems. This powder range was selected to guarantee an optimized result for implementation with the “PX” range and the former “PM” range systems produced by Phenix Systems. [1-86]

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The Co-Cr alloy powder (ST2724G) used for DMLS manufacturing presents the chemical composition: 54.31 %Co; 23.08%Cr; 11.12% Mo, 7.85% W, 3.35% Si, and Mn, Fe < 0.1%. [35-41]. Table 7 presents the mechanical characteristics of the Co-Cr powder used for the DMLS manufacturing process.

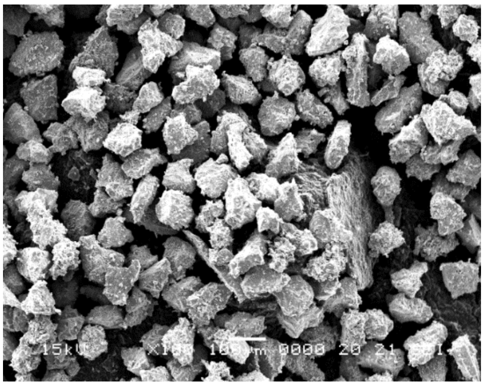

Figure 11. Ti6Al4V powder obtained by hydride-dehydride

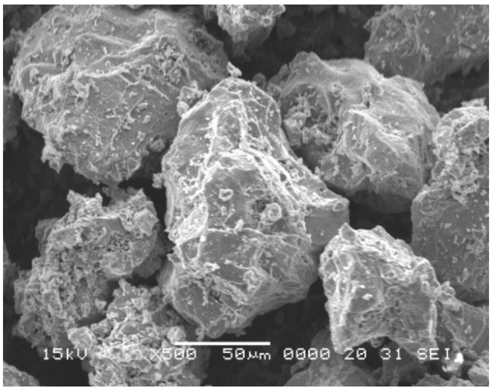

Figure 12. Compact and irregular Ti6Al4V grains

The main mechanical characteristics of Ti6Al4V powder are: elastic limit 0.2% $Rp_{0,2}$= 815 MPa, elongation at break = 10%, Vickers hardness = 375 HV , elastic modulus = 229 GPa, mass density = 8,336 g/cm^3, corrosion resistance < 4 µg/cm^2, and thermal expansion coefficient = 14,5×10^{-6} K^{-1}.

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The morphology investigation and semi-quantitative analysis of powder were performed using a QUANTA INSPECT F scanning electron microscope and x-ray spectrometer for energy-dispersive (EDS) with a 133 eV resolution at MnK.

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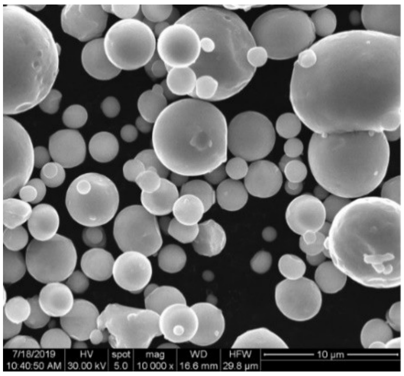
The DLMS process consists of sintering a powder material using a laser that binds powder grains to materialize a solid structure by welding. The un-sintered or loose material is removed after the sintering process is finished on the machine and can be recycled for future use, making it both economical and environmentally friendly.

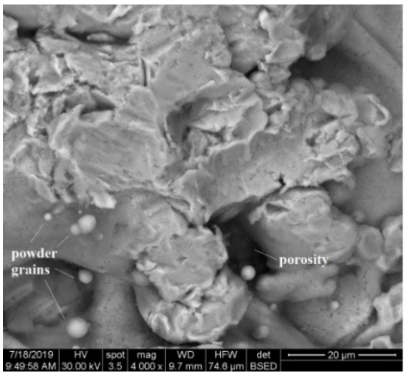
Table 7. Mechanical characteristics of Co-Cr powder

Minimum layer thickness	20 µm
Surface roughness	Ra=10 µm, Ry=40-50 µm Ra=0,39 µm, Rz=1,6 µm After polishing Rz<1 µm
Density with standard parameters	8,3 g/cm^3
Mechanical properties	
Tensile strength	1100MPa
Yield strength	600 MPa
Elongation at break	20%
Young's modulus	200 GPa
Hardness	35-35 HRC
Fatigue life	>10 million cycles
Thermal properties	
Maximum operating temperature	1150 ^{0}C

The Co-Cr alloy powder has low granulometry, and the spherical grain size is approximately 20 µm, as shown in Figure 13a). Figure 13b) shows the DMLS sintered structure of a dental part; this image also shows the specific porosity. The EDAX analysis of the Co-Cr powder is illustrated in Figure 14.



a) (x10000)



b) (x4000)

Figure 13. SEM analysis of a) Co-Cr powder; b) DMLS sintered structure

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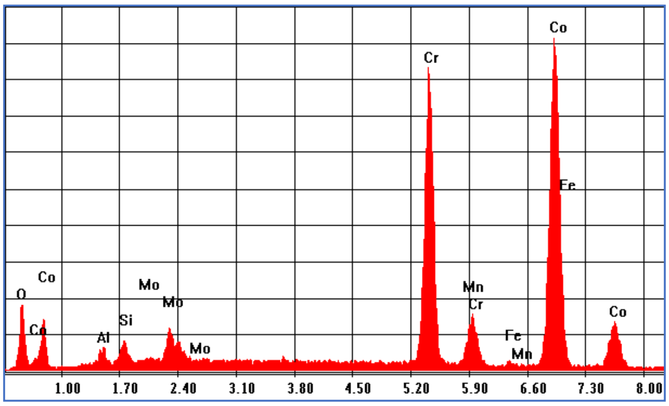

Figure 14. EDAX analysis of Co-Cr alloy powder

Figure 15. Analogue dental implants manufactured by DMLS

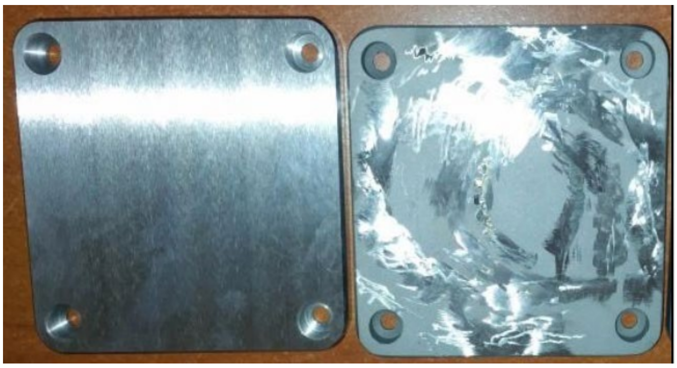

Figure 16. The platen of sintering machine

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In figure 15 are presented analogue dental implants manufactured by DMLS using Co-Cr alloy powders and a Phenix Systems machine. The Phenix System machine has a rectangle platen used in the sintering process, as in figure 16.

The material used for the platters is martensitic stainless-steel Fe-15%Cr, with magnetic properties. It has excellent mechanical properties (strength, hardness), corrosion resistance and heat resistance. The spark type is fragmented, straight, white characteristic of martensitic stainless steels. Platens made of martensitic stainless-steel Fe-15%Cr allow to be reused, by carrying out milling and rectification operations, necessarily requiring a hardening treatment to increase the hardness of the superficial layer on the surface of the platen. In figure 17 is presented the SEM analysis of Co-Cr alloy chips and stainless-steel martensitic Fe-15%Cr of the platen of the machine. [1-86]

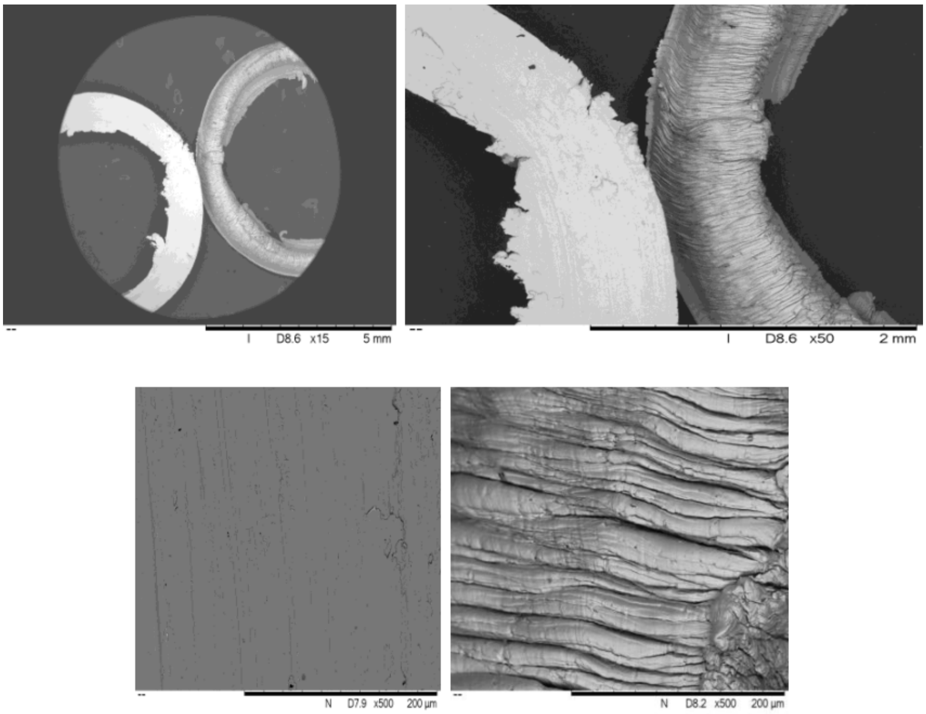

Figure 17. SEM analysis of Co-Cr alloy chips and stainless-steel martensitic Fe-15%Cr of the platen of the machine

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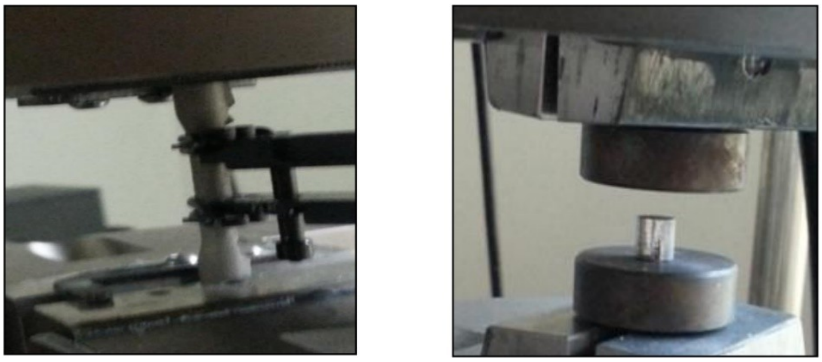
To determine the tensile and compressive strength (Fig. 18) was used an INSTRON 8810 machine, having the speed of 0.5 mm/min. The curves obtained at traction and compression show excellent mechanical resistance, maximum tensile that the samples withstand is 1200 MPa, being a superior mechanical resistance that is required in medical applications (dental crowns or analogue dental implants), as in the figure 18. [1-86]



a) b)

Figure 18. a) Traction and b) Compression tests of Co-Cr alloy manufacturing by DMLS, using INSTRON 8810 machine

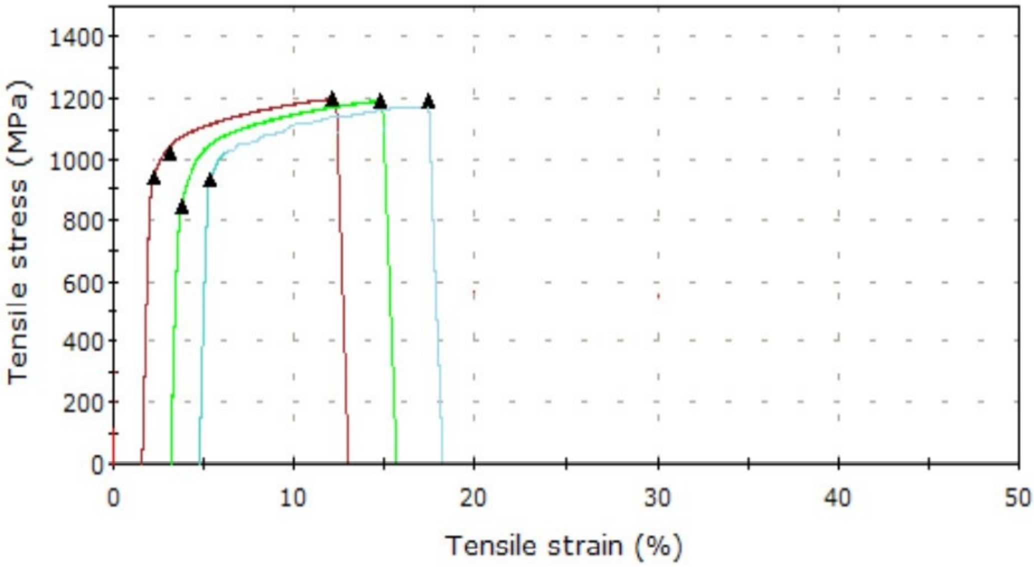


Figure 19. Traction curves obtained for the Co-Cr alloy manufactured by DMLS

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It is noted in the case of tensile tests that the samples break suddenly perpendicular to axis of the specimens, around the stress of 1200 MPa and we can notice the behavior mechanically strong brittle (Fig.19), specific to sintered metallic powder through SLS and DMLS technologies. [1-86]

The morphological structure of the alloy powder tensile specimen of Co-Cr sintered by DMLS technology is shown in figure 20 and can observe the deformation of the material, the structure with deformed pores and the appearance of cracks in the material and regular layers.

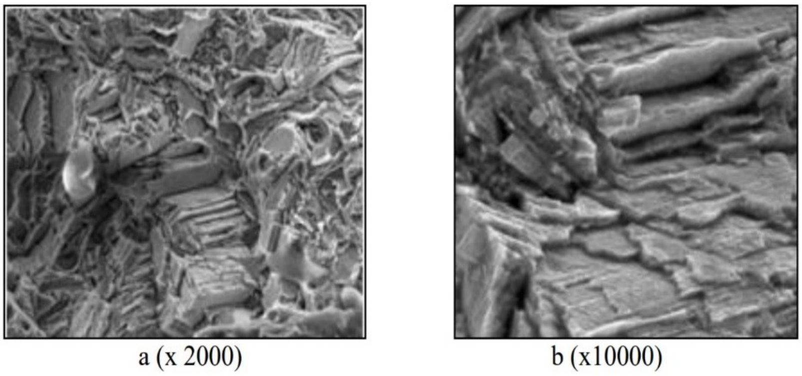

Figure 20. SEM analysis of the samples of Co-Cr superalloy after the traction tests

In the case of compression tests, the behavior is also noted mechanically strong brittle, specific to sintered materials from metallic powders, as in fig. 21.

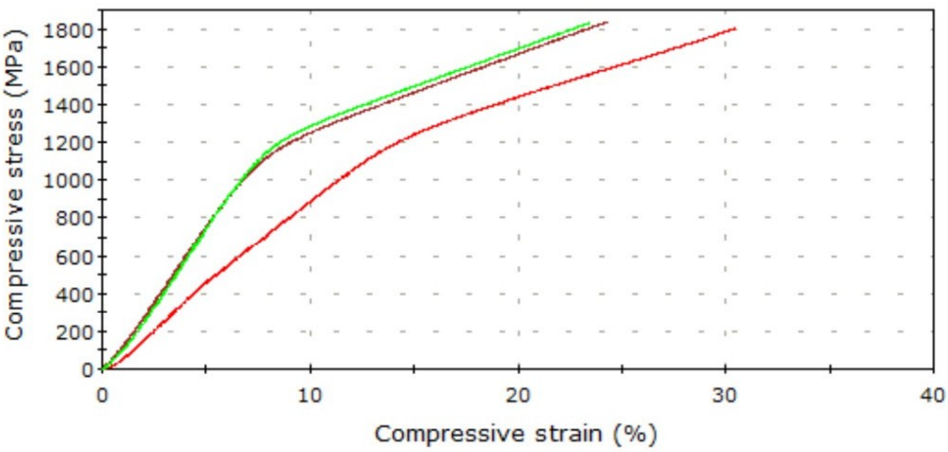

Figure 21. Compression curves obtained for the Co-Cr alloy manufactured by DMLS

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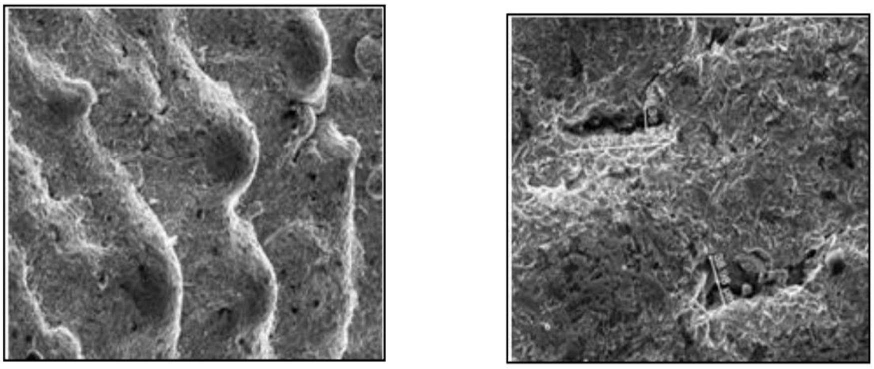
Also, the mechanical strength is up to around 1200MPa. The specimens became barrel-shaped, the deformation is accentuated with deformed pores, regular layers are also noticeable, as in Fig. 22.



a (x400) b (x1000)

Figure 22. SEM analysis of the samples of Co-Cr superalloy after the compression tests

Following tensile and compression tests it was noted that the samples sintered from Co-Cr alloy powder and obtained by DMLS technology, present excellent resistance up to around 1200 MPa, this resistance is a lot superior to that required in medical applications. [1-86]

The ICP-MS analysis (Inductively Coupled Plasma – Mass Spectrometry) was used for determining the amount of metal ions dissolved by mass absorption from SBF in which 6 Co-Cr samples sintered DMLS, were subsequently immersed in SBF – Simulated Biological Fluid (3 samples supported a post sintering treatment in the furnace, at 800°C, during for 30 minutes). The instrument used was an ICP-OES spectrometer 725.

Table 8. ICP-MS analysis

Samples	pH
S1 thermal post-treatment	7,77
S2 thermal post-treatment	7,76
S3 thermal post-treatment	7,74
S4 without treatment	7,74
S5 without treatment	7,73
S6 without treatment	7,77

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In table 8 are presented the ICP-MS analysis, the determination of the metal ions of Co, Cr, Mo, W was carried out with ICP-OES device model 725, from Agilent Scientific.
For all samples the amounts of Co, Cr, Mo, W were < 0.0001 g/l, the amounts of metals were below the detection limit, so this superalloy can be used safely for temporary dental implants and dental crowns manufacturing.

4.1. Laser types used in Additive Manufacturing

Fiber lasers are up to 100 times more powerful than CO_2 lasers and are also used for sintering metal powders. The diode has a long life.
Fiber lasers are the most reliable and most used in industry.

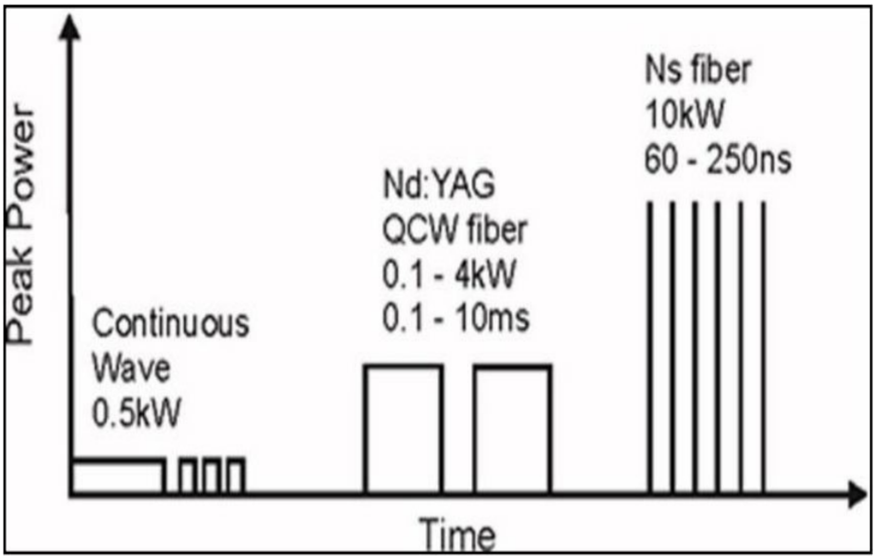

Figure 23. Differences between YAG and fibers lasers

Crystal YAG lasers belong to semiconductor lasers and are suitable for processing metal powders, having the same wavelength as 1,064 micrometer fiber lasers. But they have a short life and are very expensive. [1-86]
The difference between YAG and fiber lasers is the difference in values laser power, as in figure 26. Metals like Al, Ag, Au which are very heat-conducting and highly reflective, present a high thermally gradient and during the manufacture of the powder can appear the agglomeration,

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a powder conglomerate around of the sintered part, a phenomenon noted in the case of aluminum. So, the pieces will not be well delimited. In figure24 are presented different laser modes.

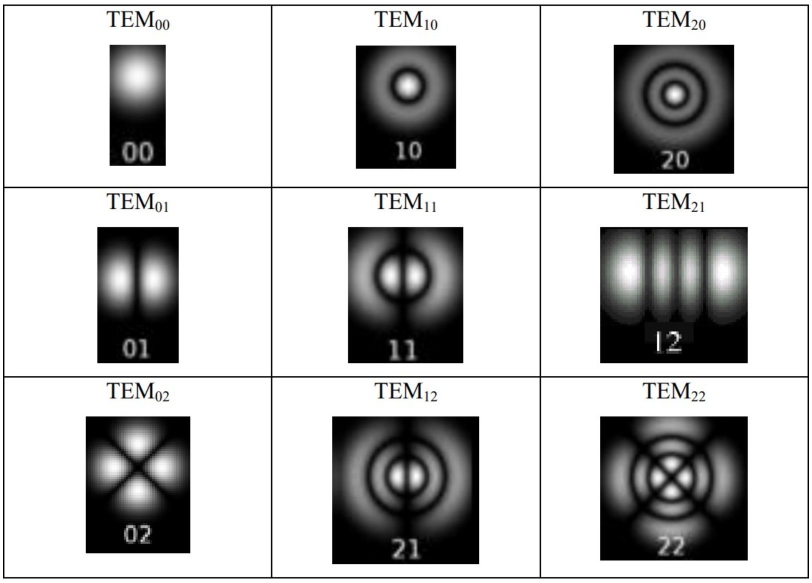

Figure 24. Different laser modes

Fiber lasers can be:
-monomodal, the diameter of the core is very small of 9 µm, the outer diameter is
of 125 µm and presents a single mode of light propagation (9/125) – fig.25;
-multimodal, because light propagates in several ways, so it can follow several trajectories inside the core and are of the type (50/125 or 62.5/125 in µm) – fig.26.

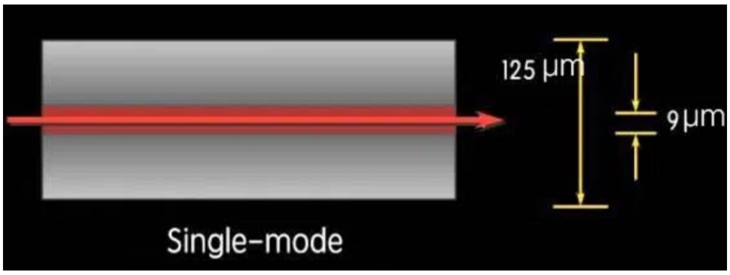

Figure 25. Monomodal laser fibers

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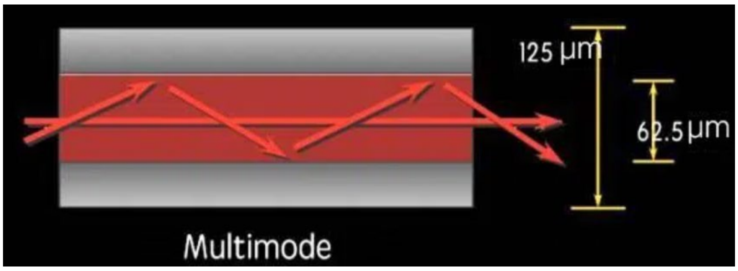

Figure 26. Multimodal laser fibers

For example, the PM100 manufacturing equipment from Phenix Systems (fig.27) uses a powder with a very fine particle size of 10 µm, a laser Nd:YAG power of 200W, the laser beam is of monomodal type TEM00 showing a Gaussian-like power density profile.

The LF 250 manufacturing equipment from TrumaForm (fig.28) uses a powder with a grain size greater than 30 µm, the laser beam is of the type multimode TEM10 showing a Flat-Top power density profile.

Figure 27. Phenix Systems – PM100 system

Figure 28. TrumaForm LF250 system

In the case of SLM manufacturing systems, they can be equipped with a laser source, with two laser sources (Twin laser sources), respectively four laser sources (Quad) working independently, to increase productivity and reduce manufacturing time.

Figure 29 shows the Nd:YAG laser beam profiles: Gaussian laser beam, multimodal laser beam, top-hat laser beam. The most widely used in laser additive manufacturing is the "top-hat" Nd:YAG laser beam.[1-86]

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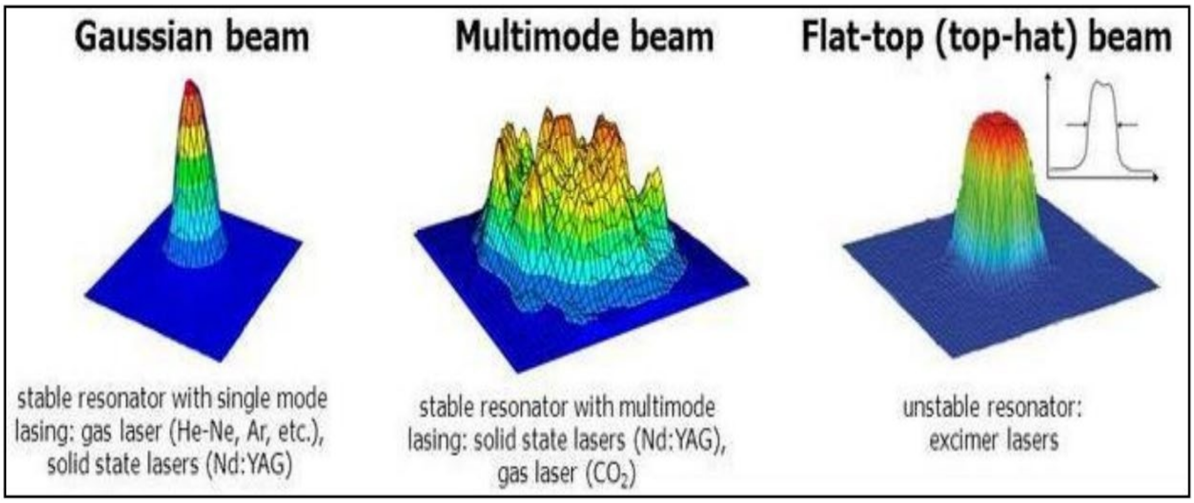

Figure 29. Laser beam profiles Nd:YAG:.a) Gaussian beam; b) Multimode beam; c)flat-top (top-hat) beam

Electrons Beam Melting manufacturing is an additive manufacturing process that uses electrons beam for metallic powders melting. This process was researched and developed by Charlmers University in Sweden in the 1990s.

Figure 30. Trabecular Lattice structures for enhanced osseointegration

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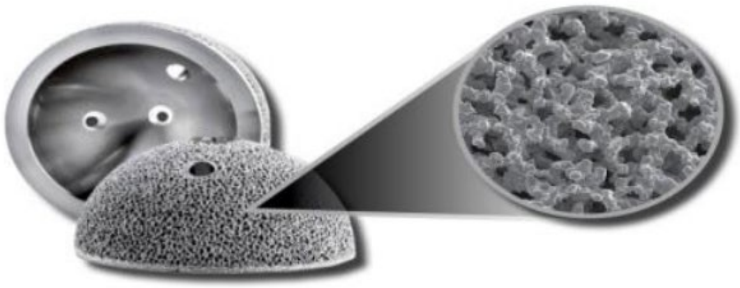

Figure 31. Acetabular cups, manufactured by EBM technology, for hip replacements, with trabecular structure, Courtesy Arcam

The raw material used in this process is metal powder of various qualities, being similar to the selective laser sintering process SLS and selective laser melting SLM. Specific to the EBM process is the fact that the laser is replaced by electrons beam whose power can reach 4kW, as a melting source.

The materials frequently used by this process are pure commercial Ti, Ti-6Al-4V alloy, the Co-Cr alloy, Inconel 718 and Inconel 625. This process allows the development of the manufacturing parameters in order to make parts from alloys of Cu, Nb, Al 2024, stainless steels, titanium aluminide.

The process consists in projecting a beam of electrons that reaches half the speed of light, on the surface of the powder bed, and their kinetic energy is transformed into heat, melting the metal powder.

The structure of the material manufactured by EBM technology is unique, trabecular, i.e. with a very small porosity of the order of microns.

The EBM trabecular structure allows optimization of osseointegration due to pore geometry, pore size, relative density and roughness.

The metal implant with trabecular structure has become the preferable treatment, solving difficult cases and greatly increasing the success rate. The implant has a composition of titanium and tantalum. [1-86]

For these patients, studies highlight different trabecular lattice structures obtained by EBM and that permit the appearance of neoformation bone in and on the surface of the implant in

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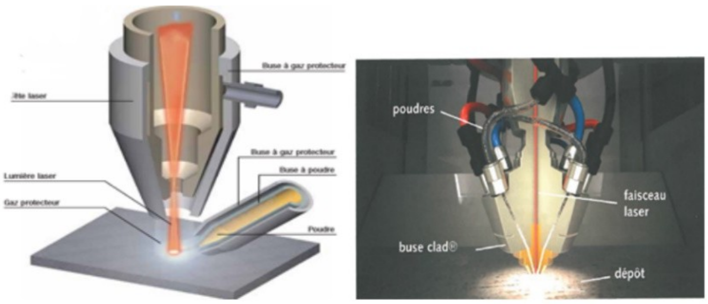
the first 2 weeks after implant insertion, as in figure 30. In figure 31 is presented an acetabular cup, manufactured by EBM technology, for hip replacements, with trabecular structure

4.2. Metallic powders and filaments used in Laser Metal Deposition and Direct Metal Deposition

In the field of powder projection for the technology Laser Metal Deposition (LMD), two techniques exist: the powder can be injected laterally (Figure 32(a)) or coaxially to the laser beam (Figure 32(b)). The first technique, very simple to manufacture, involves difficult serving of the position of the injector in relation to the movement of the nozzle to achieve complex shapes.
This explains the use of coaxial nozzles in most cases for multi-directional reloading. This second technique also allows a better homogeneity of the deposit, due to longer irradiation of the filler material and regular. Finally, side spray nozzles are often more cumbersome. Considering their bounding volume when programming trajectories manufacturing is more delicate. [1-86]



(a) Buse latérale de Kennametal (b) Buse coaxiale de BeAM

Figure 32. a) Laterally Kennametal buse; b) Coaxially BeAM buse

Several studies have been carried out to compare laser cladding technology and TIG (Tungsten Inert Gas). In most cases, these studies are carried out in order to promote laser reloading, it is thus difficult to find arguments against laser reloading.
The precision of the laser compared to TIG welding makes it possible to produce parts complex, requiring less re-machining as shown in Figure 33.

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Concerning laser cladding, the heating zone being more localized, the heat affected zone (HAZ), which is the site of metallurgical modifications of the metal of base that can induce fragilities, reductions in mechanical resistance, lack of ductility is lower. For the same reasons, the deformations induced in the part by thermal increase are less important.

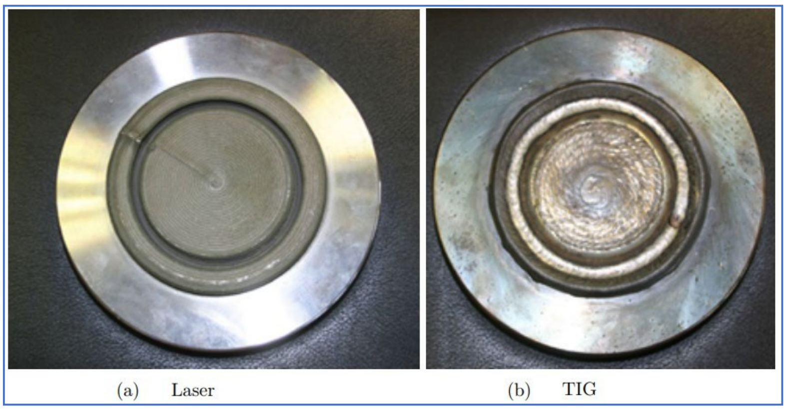

Figure 33. Geometry resulting from LMD process and TIG welding

Another advantage of laser cladding is the lower dilution area compared to in TIG welding, this small dilution zone makes it possible to obtain a homogeneous deposit and having the desired characteristics throughout its thickness, unlike the TIG welding which presents a strong dilution, degrading the quality of reloading and an uneven fusion depth. This feature is illustrated in Figure 34. [1-86]

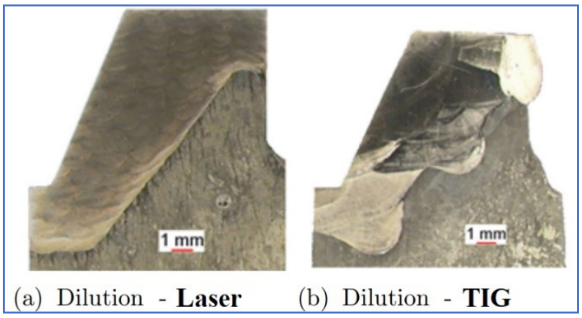

Figure 34. ZAT after: a)LMD process and b)welding TIG

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Microstructure analysis reveals a finer structure in the case of the laser cladding than in the case of TIG welding. The concentration of energy in the case of the laser makes it possible to obtain a higher cooling rate, which reduces the grain size and therefore increases the hardness of the material. In the case of a stellite 6, can notice a hardness of 48-50 HRC against only 40-42 HRC in the case of welding TIG. Figure 35 shows these differences in microstructure. [1-86]

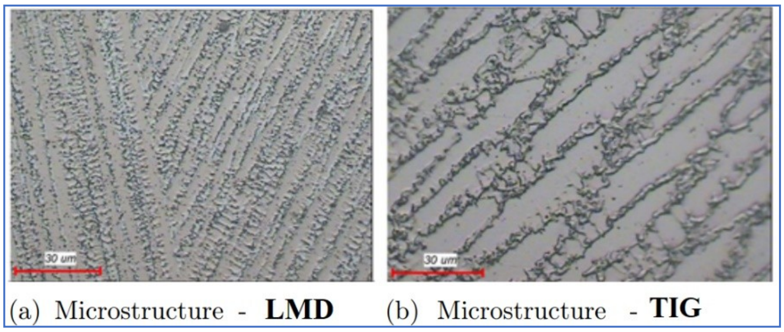

Figure 35. Microstructure after: a)LMD process and b)welding TIG

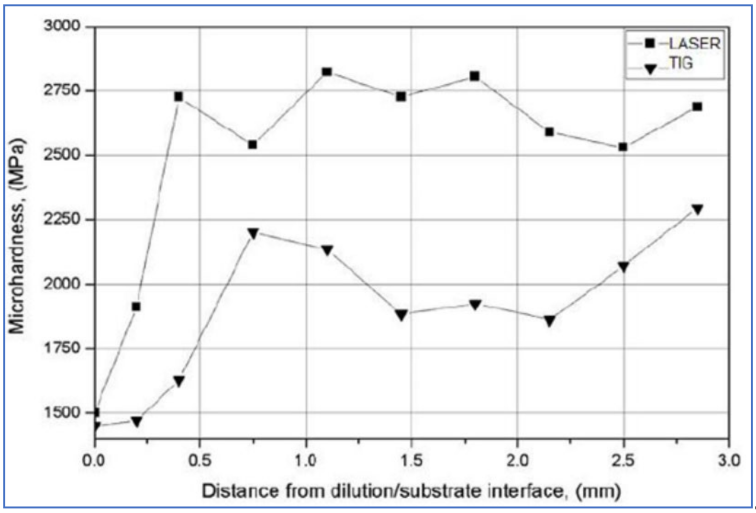

Figure 36. Evolution of the microhardness as a function of the distance to the hardened zone for an Inconel 625 coating added by laser or TIG

The TIG process, like MIG, is characterized by a significant increase in temperature in the room, which leads to recrystallization and weakening of the base alloy in the working area, as can be seen in the graph of Figure 36.

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4.3. Architectural materials manufactured by SLM

In figure 37 are shown simple geometric cells for architectural materials, manufactured by innovative ALM (Additive Laser Manufacturing) technologies.

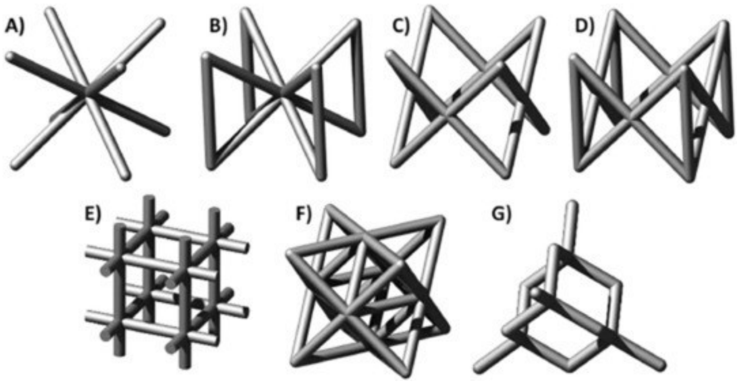

Figure 37. Strut-based lattice structures: BCC (A), BCCZ (B), FCC (C), FCCZ (D), cubic (E), Octet-truss (F), and diamond (G)

The most common strut-based cell topologies that have been investigated are body-centred cubic (BCC) and face-centred-cubic (FCC), or variations of these, such as the inclusion of z-struts (BCCZ and FCCZ) (Fig.39) which are named after analogous crystalline structures. Other strut-based topologies also exist, such as the cubic, octet-truss and diamond.

These topologies are generated using mathematical formulae that define the U ¼ 0 iso-surface boundary between solid and void sections of the structure (Table 9) [46]. Various parameters such as periodicity and relative density can be altered to tune their mechanical performance. Two industries which have taken particular interest in AM lattice structures are the biomedical and aerospace industries. [1-86]

AM lattice structures are used in the biomedical industry for medical implants, the global market for which is expected to grow to $116 billion by 2022. The ability to produce high-quality metallic components that conform to complex, patient-specific surfaces makes SLM perfect for the fabrication of medical implants, and the ability to produce metallic components with stiffness closer to that of bone makes AM lattice structures perfect for biomedical applications.

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These structures can be designed to produce optimal osseointegration and have been shown to sustain excellent bone in-growth and achieve high performance in terms of implant fixation. Beyond the manufacture of metallic implants, potential innovative design applications of AM lattice structures have been suggested, such as Burton et al. who have proposed an implant which incorporates a reservoir that locally releases a therapeutic drug to achieve antimicrobial functionality by incorporating a reservoir. [1-86]

The light-weighting potential of AM lattice structures, such as the replacement of internal solid volumes with lattice structures with a similar strength, means AM is of great interest to the aerospace industry. Other properties of AM lattice structures, such as the ability to produce conformal cooling channels also makes them an attractive option for aerospace applications. Zhou et al. have developed a lightweight phase-change thermal controller based on lattice cells [85]. Thermal controllers are an important component to manage the temperature of various electronics in spacecraft, yet traditional designs considerably add to the spacecraft's weight. Using an SLM-fabricated lattice sandwich structure Zhou et al. were able to produce a thermal controller with 50% increased thermal capacity compared to traditional alternatives with similar mass. In the table 9 are presented the SLM processing parameters and in the table 10 are presented the properties of metallic materials used in SLM technology.

Table 9. SLM processing parameters [82]

Machine	Manufacturer	Material	Spot size (μm)	Border power (W)[a]	Hatch power (W)	Scan speed (mm/s)	Hatch spacing (mm)	Layer thickness (μm)	Mean powder size (μm)
ProX DMP 300	3D Systems	Ti-6Al-4V	–	–	–	–	–	–	8.64
ProX-300	3D Systems	SS 630 (17-4PH)	70	170	170	1600	0.05	40	–
Concept X-line 1000R	Concept Laser Company	AlSi10Mg	–	–	370	1500	0.19	30	31
M2 Cusing®	Concept Laser Company	Ti-6Al-4V	60	150	150	1750	0.075	20	20–50
EOSINT-M270	EOS	Ti-6Al-4V	100	58.5	117	225	0.18	30	45
M280	EOS	316L SS	100	–	–	–	–	–	20–40
DMLSEOSINT-M270	EOS	Ti-6Al-4V	100	170	170	1250	0.06	30	20
M 270	EOS	Ti-6Al-4V	100	170	170	1250	0.1	30	29
M 270	EOS	Ti-6Al-4V	10	117	117	225	0.18	30	–
M280	EOS	AlSi10Mg	–	370	370	1500	0.13	30	30
Realizer II	MCP	316L SS	90	80–160	80–160	–	–	–	16–38
MCP Realizer 2, 250 SLM	MCP	Ti-6Al-4V	54	80	80	–	–	50	45
Realizer SLM Workstation	MCP	316L SS	40	95	95	–	0.075	75	45
AM250	Renishaw	AlSi10Mg	80	200	200	–	0.13	25	–
AM250	Renishaw	Ti-6Al-4V	–	100	200	–	0.065	30	–
AM250	Renishaw	AlSi10Mg	70	–	–	–	–	25	–
SLM 125	SLM Solutions	CP-Ti	–	100	100	385	0.12	–	36.6
SLM250HL	SLM Solutions	Ti-6Al-4V	–	100	100	375	0.12	30	40

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Table 10. Properties of metallic materials used in SLM technology [82]

Material	Property	Value
Inconel 625	Density	8.44 g/cm^3
	Yield strength	460 MPa
	Modulus	205.8 GPa
	Density	4.43 g/cm^3
TiAl64V	Yield strength	880 MPa
	Modulus	193 GPa
	Density	8 g/cm^3
Stainless steel	Yield strength	205 MPa
	Modulus	193 GPa
	Density	2.67 g/cm^3
AlSi10Mg	Yield strength	240 MPa
	Modulus	70 GPa

Table 11. Quasi-static compressive test data for SLM lattice data collected from literature, ordered by unit cell topology [82]

Topology	Material	Relative density (%)	Cell size (mm)	Strut diameter (mm)	Geometry (N = nominal, M = measured)	Compressive strength (MPa)	Modulus (MPa)	R
BCC	316L SS	3.5–13.8	1.25	0.19–0.22	N	0.36–5.89	17.89–378	
BCC	316L SS	2.3–4.3	–	0.162–0.23	N	0.2–1	8.68–57.56	
BCC, BCCZ	316L SS	5.3–16.6	1.5–2.5	0.25	N	0.92–15	50–2700	
BCC, BCCZ	AlSi10Mg	0.7–22.2	10	1	N, M	0.46–4.36	21.71–490.22	
BCC, BCCZ, FCC, FCCZ	Inconel 625	2.5–13.8	2–4	0.3	N, M	0.8–10.9	22.1–1246	
BCC, BCCZ, FCC, FCCZ, FBCCZ, FBCCXYZ	Ti6Al4V, AlSi12Mg	7.5–39.7	2–7.5	0.3–1	N, M	4–124	110–2780	
BCC, Octet-truss	SS 630 (17-4PH)	43	–	–	N, M	–	9710–13,960	
Diamond	Ti6Al4V	3.6–26.5	–	–	N	8.2–99.64	370–4240	
Dodecahedron	Ti6Al4V	15.78–314.55	–	–	N, M	19.4–117.2	550–3490	
Gyroid	CP titanium	26.7–31.3	2–3	–	N	44.9–54.5	1465–2676	
Gyroid, Schwartz diamond	Ti6Al4V	5–95	3–7	–	N	47–1559	120–17,190	
Octet-truss, Rhombicuboctahedron	AlSi10Mg	10.4–14.7	–	–	N, M	4.7–9.1	690–1250	
Schwartz primitive, cylinder grid	Ti6Al4V	70–90	1.5	0.37–0.58	N, M	–	920–2420	
Simple cubic	Ti6Al4V	19.4–36.2	–	0.54–0.64	N, M	108–170	5360–8730	
Truncated cuboctahedron, simple cubic, diamond	Ti6Al4V	20–34	–	–	N, M	31.7–112.6	2180–4578	

In Table 11 are presented the quasi-static compressive test data for SLM lattice data collected from literature ordered by unit cell topology.

Other areas of study which would facilitate the greater characterization and commercial adoption of SLM lattice structures include:

-The performance of SLM lattice structures have been characterised in their fabricated state, but knowledge of their performance during operation is still limited. The thermal behaviour of SLM lattice structures during operation, especially the effect of atmospheric phenomena for aerospace applications, and the effect of biodegradation on the fatigue strength of SLM lattice implants for biomedical applications are yet to be characterised. [1-86]

-Most research on SLM lattice structures focuses on either strut-based or TPMS lattice structures discretely. Research on their comparative performance is still needed to identify the appropriateness of these design approaches for different applications
and performance requirements.

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-The tensile performance of SLM lattice structures is still not well defined as most research uses compressive experiments for identification of their mechanical properties largely due to the greater difficulty of tensile lattice experimental design.

Knowledge of the behaviour of lattice structures is quite disparate, as researchers have investigated isolated areas of the broad field of lattice structures. Greater homogenisation and standardisation of this field could lead to new insights and enhance the applicability of SLM lattice structures for all applications. SLM allows the fabrication of complex geometries with high resolution, which is perfect for the manufacture of lattice structures. Using this technology, lattice structures with highly tuned geometries and topologies can be fabricated to produce a wide variety of properties unachievable by their bulk materials. This has led to research into these structures, particularly for biomedical, light-weighting and energy-absorption applications. However, there exists no overarching analysis of this data.

In figure 38 are presented the mechanical properties of the principals alloys used in Additive Laser Manufacturing. [1-86]

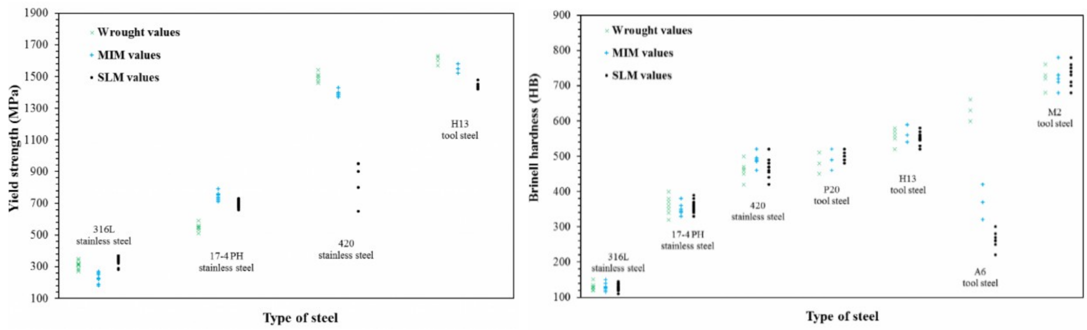

Figure 38. Mechanical properties of the principal's alloys used in Additive Laser Manufacturing

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