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Metal Additive Manufacturing: Process, Conception and Post-treatments

MEMÒRIA

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Abstract

This research project is a global study of the metal additive manufacturing from the optimization of the design phase to the improvement of the final state of the parts with finishing treatments and passing by the adjustment of the production process.

It starts with a state of art of the technologies, materials and industrial projects leading to focus on the laser melting of a metallic powder bed.

The study of the manufacturing process has been realised focusing on the laser’s parameters: its speed and power. The optimization has been made with the objective of improving the surface state of the parts as long as the production was concerning dental implants in Cobalt-Chromium alloy.

The design phase has been focused on with the topological optimization method applied to a suspension’s triangle in stainless steel. This method leading to a reduction of the mass and/or gain of stiffness of the part is used a lot with the additive manufacturing as long as it permits complex geometries. It although has to be linked to real tests to get information like the mechanical characteristics necessary for the simulation and to validate or not the resistance of the new geometry.

The improvement of the final state of the parts can be related to the surface aspect with the shot peening process but although to the internal state of the part with post heat treatments. Here various heat treatments have been realised on a stainless steel, an aluminium alloy and a Nickel based superalloy to reduce the internal residual stresses and improve the homogeneity of the parts. Different macro and microscopic characterisations have been realised to validate or not the effect of the treatments on the quality of the samples to avoid their deformation and break and to try to approach the characteristics of a part issued from a more classical metal manufacturing process.

Keywords: Metal Additive Manufacturing, Selective Laser Melting, Direct Metal Laser Sintering, Topological Optimization, Post Heat Treatments
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Introduction

This master thesis internship has been realised in the company Ecosteering, subsidiary of Ennodev based in Plaisir and implementing industrial projects. Ennodev supports clients in strategical analyses, R&D and industrial management and revitalization of industrial sites by transition management.

The additive manufacturing appeared in the late eighties opposing the more conventional processes like the machining of material blocs. In fact, with the additive manufacturing the principle is the adding of the material layer by layer using as model a numerical design when the other processes remove material with milling for example. Thus it permits to access to parts with complex geometries, customizable and simplified in their assembly. These parts can be topologically optimized and thus be lighter and better heat conductors.

It’s the company 3DSystems that initiated this new way of producing and the rapid prototyping era with its stereolithography technology using liquid and photosensitive epoxy resins polymerised by laser. This technology allows to obtain more conceptual parts than functional with their good surface state but poor heat and mechanical resistances. The rapid prototyping enables to improve the competitiveness and final value of a product thanks to its impact on the time, the quality and the cost of the production. The development time can be reduced by 25% and the cost by 50% avoiding the use of tools and the process of certain heat and surface treatments. The rapid prototyping gives to the companies the opportunity to stay innovative as well as the time to market is reduced. The parts can be produced on demand for reparation need for example enabling to reduce the stock: the continuous production could then be reduced.

This new manufacturing process is now turning more rentable for the industrials, the produced parts gain in resistance allowing to be used for more than just the prototyping phase. It’s now possible to produce functional parts that are used in the production and post-production phases of the product lifecycle. From the rapid prototyping concept appeared the rapid tooling and rapid manufacturing ones. Despite of this, the development of the manufacturing systems is still necessary to be able to adapt them to large productions with a better repeatability, a larger choice of materials and better surface states. The research focuses on the production of parts with precise geometries and low internal stresses’ states close to the one possible to reach with conventional manufacturing processes.
The following project focused on the Metal Additive Manufacturing with a study of the different existing processes and material used in the main industrial projects. Then, after choosing a precise technology, an optimisation of the manufacturing process has been realised as well as the improvement of the parts’ conception intended to it. Post-treatments have although been analysed in the following part with the objective of improving the quality and homogeneity of the produced parts.

The characterisation phase of the metal additive produced parts has been realised with the Technological Resources Centre Analyses&Surface based in Val-de-Reuil. This laboratory realises all types of materials’ studies thanks to its characterisation tools and climatic chambers enabling to reproduce the ageing of the parts. In this way, it supports its clients in their R&D projects. Analyses of failures are although made as metallurgical expertise, corrosion phenomenon studies and defects analysis as foreign bodies.

The samples analysed at the TRC have been produced by Initial, a company based in Seynod and French leader in the development of additive manufacturing products.

The optimisation of the metal additive manufacturing process has been realised with 3D Dental Store based in Rouen. This company develops and commercialize 3D systems for dental application like scanners enabling the digitization and modelling of 3D implants. It although manufactures implants and prosthesis for its clients.

The topological optimization simulation has been finalised and validated with the Centre d’Essai en Vibro-Acoustique pour l’Automobile based in Saint-Etienne du Rouvray and working on the numerical simulation and mechanical calculations to improve the resistance or optimize the design. The mechanical tests necessary to bring the numerical data for the calculation have been realised in the Laboratoire Havrais d’Essais Mecaniques based in Gonfreville-l’Orcher.
Chapter 1: State of art

1 The manufacturing processes

Nowadays, the materials used in the additive manufacturing processes are under the form of powders, filaments, sheets and liquids in plastic, metal, ceramic, paper, wood, wax, composites or bio materials.

The different processes are:

- The extrusion of filaments like the fused deposition modelling which is the most known and less expensive technology.

![Figure 1: FDM (CustomPart)](image1)

- The photopolymerization in tank like the stereolithography which is the oldest process.

![Figure 2: Stereolithography (CustomPart)](image2)
The deposition of liquid materials as solidified photopolymers with UV treatment or the projection of binder on powder bed to fuse the particles

Figure 3: Deposition of liquid materials: UV treatment (left) and projection of binder (right) (CustomPart)

The laminated objected manufacturing in which the paper or metallic sheets are laser cut and bound with a water-soluble adhesive in the case of the paper and ultrasounds in the case of the metal.

The melted powder bed enabling to create parts with polymers or fused metals with laser or electrons beams.

The material deposition using metal powder or filament melted during the deposition under laser or electrons beams.

The three last processes using and more precisely the ones using metal powders are the most interesting ones for large-scale development of the additive manufacturing in the industry.

2 The Metal Additive Manufacturing

2.1 Melted powder bed

2.1.1 Laser beam

This process uses as material a metallic powder deposed on a platform moving vertically and as energy source a laser beam going from 50 to 2000W.
A recoater arm spreads the powder on the platform in uniform layers from 20 to 200 micrometres and then the computer-controlled energy source, targets the powder following the layer geometry to melt the powder and solidify the part. The platform, then, lowers of the specified layer's depth and a new quantity of powder, coming from the supply is spread.

The manufacturing process is realised in a chamber under inert neutral gas, Argon, Helium or Azote because of the very sensitive metallic powders that can easily oxidize. It’s for this same reason that the storage of the powders is a really important parameter in the metal additive manufacturing process. The humidity and the oxygen quantity have to be controlled to conserve the materials' proprieties while working for example with titanium, aluminium, steel or superalloys powders.

The good quality of the final part depends although on the edification supports that have to be many and robust to avoid the curving of the part for example. At the same time, it’s interesting their quantity to limit the additional cost represented by their removal process and the material used and lost for their manufacturing process. They are still necessary as long as the chamber’s temperature is away from the powder’s melting point. Thus, material removals and mechanical stresses can occur and these ones have to be reduced to obtain final parts with a high dimensional precision.

As the material is a powder, it’s necessary to well characterise and control it. Many measures exist to get information about the grains’ sphericity, their size distribution, their chemical composition, their porosity, their pourability and their size between 10 and 50 micrometres. The choice is then made for the layer’s depth: the smaller it will be the better the part will be defined but the manufacturing duration will although increase. The reduction of the process duration is then possible by increasing the layers’ depth while still avoiding the stair effect that occurs when it’s too important and the layers are visible when the surface is curved.
The Nd YAG (Ytritium-Aluminium-Grenat) laser of a wavelength of 1060nm although has its parameters that have to be controlled. There is, for example, the power, the speed and the sweeping step from 30 to 400 micrometres. Generally speaking, the increase of these parameters allows a decrease of the porosity and it’s possible to adapt the manufacturing speed between 1 to 10 cm³/h.

The lasing strategy is although a primordial choice for the additive manufacturing: it allows to reduce the most important defects of the 3D printed parts. The crossed strategy at 90 or 67° allows to limit the anisotropy of the mechanical characteristics when the one in checkerboard limits the residual stresses in the massive parts.

![Lasing strategy](image)

*Figure 5: Lasing strategy (Techniques de l'Ingénieur)*

As most of the Metal Additive Manufacturing processes, the surface or heat post-treatments are often necessary like the shot peening, the polishing or the finish machining to eliminate an added thickness improving the precision. A heat treatment, under controlled atmosphere, is often necessary to eliminate the internal stresses and avoid its break or deformation when exiting the machine.

### 2.1.2 Electrons’ beam

This process, less widespread than the laser’s one, uses an electrons beam of 60kV and 3kW and compared to the laser’s using process it needs a conductive material. The powder supply although needs a preheating to avoid a thermal gradient too important during the fusion and the apparition of internal stresses. The manufacturing is realised under controlled atmosphere with Helium but although in a vacuumed and heated chamber at 10^-6mbar and around 700°C. This temperature is then controlled during the cooling and can lead to parts without internal stresses and mechanical characteristics close to a forged metal.
This process can be more productive than the one using a laser as long as the electrons beam brings more energy to the powder, moves faster, around 8000m/s, and allow to work with bigger layers' depth and powder’s size, from 40 to 150 micrometres. However the resulting surface’s states with this process are not as good as the ones with the laser’s process because the beam is wider and thus less precise.

2.2 Material deposition under concentrated energy

This process brings the material as a powder jet or a filament in the melting zone close to the surface. The melting is processed by a laser beam for the powder and an electrons one for the filament.

The manufacturing is made in a chamber under controlled atmosphere with an inert neutral gas like Argon Helium or Azote. Its precision will be inferior to the Direct Metal Laser Sintering one because of the important layers’ depth used of 500 micrometres. This depth
and although the important internal residual stresses’ state obliges to realize a surface and heat post treatment to smooth the surface because of the stair effect and to relieve the tension in the part. The flow rate of the powder is around $300 \text{cm}^3/\text{h}$ with a powder’s size from 40 to 80 micrometres: thus, the manufacturing speed is more interesting than the DMLS one, reaching here $150 \text{cm}^3/\text{h}$.

This process is although interesting enabling the reparation, the function adding and the surface treatment on already manufactured parts. The supply of material is done from a nozzle moving on three axles on a platform on 2 axles: this mobility can simplify the manufacturing with the reduction of supports use for example. However the elimination of the supports use can limit the feasible geometries.

2.3 Laminated Object Manufacturing

This process uses as material metallic sheets, it’s a mix between an additive and a subtractive manufacturing process. In fact, during this process the sheets are laser cut and stuck between each other thanks to an adhesive melted with a heating compression roll. This process enables the manufacturing of big parts, it doesn’t need vacuum chambers limiting the volume. The manufacturing is not realised at high temperature which allows to include cables or various electronic components in the parts that would not support the manufacturing conditions of the metal powders’ processes. However the dimensional precision is lower than the other processes what doesn’t allow to manufacture interesting functional prototypes with complex geometries. One of the interest of the only additive manufacturing process is although lost: the reduction of material waste with the machining.

![Figure 8: Laminated Object Manufacturing (CustomPart)](image)

Another similar process exists with ultrasonic technology used to stick the sheets previously cut by milling. A solid welding is realised under pressure thanks to the vibrations around
20000Hz. This type of welding enables to mix different metallic materials in the part which don’t have the same melting temperature, something impossible with a process using a metal powder.

3 The Metal Additive Manufacturing in the Industry

3.1 Processes and materials in metallic parts’ study

To manufacture metallic parts the most used processes are the ones based on the laser’s energy. As already mentioned the electrons beam allows a faster manufacturing process thanks to the more important energy’s transfer but it although decreases its precision. It obliges too to use conductive materials what reduces the manufacturing possibilities.

Between the melted powder bed process and the material deposition one, the most interesting one is the first. Developed since longer, it now enables a better precision even if the use of several supports is necessary. The loss of material represented by the supports is not considered as a defect as long as it gives no limit to the feasible geometries.

The study of metallic parts will then focus on the melted powder bed by laser beam. With this process, two different technologies exist and are different by the type of fusion used. There is the Selective Laser Melting and the Direct Metal Laser Sintering.

The sintering process uses less energy, it doesn’t allow the full fusion of the material but brings it to a state in which the molecular fusion is possible. Thus, it permits to control the porosity of the parts depending on the level of sintering.

The selective melting gives a full and homogeneous fusion of the material but necessarily needs a precise treatment’s temperature objective, a unique fixed fusion point. This process is better than the sintering one, it enables to reduce a lot the porosities and thus to create parts with a better break resistance. The density of these parts can theoretically reach 99.9% and their mechanical properties are better than in the case of a sintering process. However it’s only possible in this case to work with mono material powders and not special alloys with different fusions points.
The leader in the distribution of machines and materials is the German producer Electro Optical Systems. Founded in 1989, this company is specialized in the direct metal laser sintering technology. As an example, it proposes three interesting materials for a comparative study of the macro and microscopic properties: the 316L stainless steel, the aluminium alloy AlSi10Mg and the nickel based superalloy Inconel 718.

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**Figure 9: EOS materials (Initial)**

The leader in the distribution of machines and materials is the German producer Electro Optical Systems. Founded in 1989, this company is specialized in the direct metal laser sintering technology. As an example, it proposes three interesting materials for a comparative study of the macro and microscopic properties: the 316L stainless steel, the aluminium alloy AlSi10Mg and the nickel based superalloy Inconel 718.
The stainless steel has an excellent resistance to corrosion and gives the opportunity to realise functional prototypes and spare or small lots parts. For example, it can be used for mechanical parts, thermal exchangers or turbines in the aeronautical industry.

The aluminium alloy has really good mechanical characteristics thanks to the silica-magnesium mix that brings it robustness and hardness. Its thermal properties are although good with its conductivity for example and it’s a really light material. It’s used for mechanical parts as the stainless steel and although for housing parts. However this material is less dense, less elastics and less heat and mechanically resistant than the 316L but its lightness is really interesting for the additive manufacturing that looks to reduce the mass of the assemblies.

The nickel based superalloy Inconel 718 is used for mechanical parts as in gas turbines for example. He got really good mechanical characteristics and an excellent corrosion and mechanical resistance.

The chrome cobalt molybdenum alloy can also be used for spare or small lots parts and fro functional prototypes. The aeronautical industry uses it for mechanical parts too. In addition, as being sterilisable, biocompatible without the presence of nickel and resistant to high temperatures and corrosion, it’s used a lot in the medical industry for personalised solutions production as prosthesis or implants.

### 3.2 The topological optimization

Changing from a conventional manufacturing process of metallic materials to their 3D print doesn’t only imply a change of the manufacturing process but although of the all designing phase preceding it. In fact, the Additive Manufacturing allows to create parts with more complex geometries, reducing the use and waste of material and the phases of assembly. The change from a one-piece part to a honeycomb internal structure gives more lightness and a better heat dissipation capacity but implies to fully rethink the design.

The topological optimization is part of this ecodesigning approach: it is more and more necessary to use it to optimize the material use. It consists in the ideal repartition of the material in a determined volume submitted to specified efforts. By discretizing with finite elements, it’s possible to optimize focusing for example on the maximum material’s reduction or the mechanical resistance. The functional analysis by numerical simulation enables to see the part’s zones where the efforts transit and to suppress the material where no stress is visible.
At the industrial level, the aeronautical and transports sectors in general have already showed the interest of this way of conception for prototypes or low and mid-volume parts. Renault Trucks is an example with its rethought engine for a better performance. The additive manufacturing allowed to reduce by 25% the mass and the number of parts: a gain of 120kg and 200 parts.

Airbus used the topological optimization on hinges to gain 10kg per plane. He although proposed an electrical motorbike concept in 2016 with its subsidiary APWORKS. This concept only weighs 35kg thanks to its 3D printed chassis in aluminium only weighing 6kg.
3.3 The industrial projects in the Metal Additive Manufacturing sector

Nowadays, the metal additive manufacturing principally concerns the automotive, aeronautical and aerospace sectors and the market forecasts announce an exponential global growth and a growing interest of the medical and paramedical sectors for these technologies.
The metal additive manufacturing touches these 3 sectors with the materials put in application. In fact, the aeronautical and automotive industries need to produce functional structural parts able to resist to uses in fatigue conditions under important stresses. Materials as Titanium or Aluminium alloys offer a great resistance, lightness and a good thermal conductivity. The nickel bases superalloys and the stainless steel are although used a lot in these sectors.

In the medical sector, the cobalt chrome molybdenum alloy is interesting for the manufacturing of prosthesis and implants as long as it’s resistant to corrosion and bio compatible. Titanium and stainless steel can although be used for the development of personalized medical solutions. That's the primary interest of the additive manufacturing in this sector: enabling the creation of parts with complex geometries and good mechanical properties. On this point this process is more efficient than a more conventional one as the casting or the molding.

In the aeronautical and aerospace sectors, Safran has already used the additive manufacturing to produce planes’, helicopters’ and rockets’ engines. For example, experiments have been made on more than 500 parts of the plane engine Silvercrest and showed all the advantages of the additive manufacturing in the reduction of the production time. The modification of a part doesn't imply the change of the tools and the molds anymore but only to work on the CAD file. Parts of the helicopters’ engine Turbomeca have been produced by selective laser melting with a nickel based superalloy after a validation phase. Injectors have been simplified with the assembly phase turning from a dozen of parts to a single one. Internal cooling functions have although been integrated.

In the automotive sector, the metal additive manufacturing applications are still limited to small lots productions with the competition vehicles and the supercars. The materials used are the ones enabling resistance and lightness as the aluminium the titanium and the superalloys. For example, the American company Divergent3D designed the Blade motorbike and the Dagger supercar with chassis partially 3D printed enabling to reduce the vehicle’s mass by 50%. In motor racing, housing parts and water or exhaust systems can be 3D printed too thanks to the thermal resistance of the used materials, until 1200°C for the superalloy Hastelloy X for example. The company Koenigsegg produced a supercar too, the One with an additive manufactured turbocompressor.
This innovative manufacturing process still only concerns limited markets and small lots productions. It’s mainly used for specific applications, for the development phases with prototypes and unique productions as personalised solutions. The perspectives are extremely interesting without manufacturing geometrical constraints comparing with a machining process. This process although offers a good reactivity during the development or reparations phases or even for a client’s specific order. Various challenges and issues are distinguishable in the metal additive manufacturing and slow down its adoption by the industrials.

At first, to efficiently substitute a conventional manufacturing process and convince the industrials, it’s obvious the additive manufacturing technologies have to be improved with their production efficiency. The machines and materials are still expensive and represent important investments. The manufacturing times are although still important particularly when a heat and surface post treatments are necessary on the parts. In addition, the manufacturing volumes are limited on the existing machines.

Another point on which studies are necessary is the process parameters optimisation in order to improve the parts' quality. This work is obviously specific to each type of production according to the expected result: mechanical are aesthetic improvement. This point is the first that is going to be seen in this project with the goal of dental implants’ surface state improvement and validation. The machines’ and materials’ producers sell optimized sets of parameters and the mechanical characteristics that are possible to reach but they can’t be universal to every applications.

A third primordial point in the way of radically changing the production’s methods is the good comprehension of the topological optimisation process. Changing the way of producing a part without redesigning its geometry with the topological optimisation doesn’t enable to fully benefit from the advantages of the metal additive manufacturing. The optimisation calculations and simulations, as being relatively new of parts issued from the additive manufacturing, necessarily need to be validated with real tests. These tests bring information about the mechanical resistant that can confirm or not the numerical results. Unlike the conventional manufacturing processes, the additive manufacturing doesn’t already have a precise database concerning the different materials’ characteristics. These ones can change depending on the specific application of the part and on the production repeatability. This point will although be seen in this project with static calculations on a steel suspension’s triangle and real mechanic tests on additive manufactured parts.
The last important point concerning the metal additive manufacturing is the research to improve the manufactured parts. It’s necessary to eliminate at the maximum their anisotropy and their homogeneity to better approach the conventional characteristics of the used materials. Heat post treatments are then necessary as, for example, a stress relieving one to reduce the internal stresses inducted by the manufacturing process. These treatments can although allow to improve mechanical characteristics or erase the manufacturing imprints with the laser’s fusion layer by layer. It’s then necessary to analyse the parts at their surface and heart to identify the defects and verify the efficiency of these treatments. This point will be the last focused on in this project with the analysis of the heat post treatments on additive manufactures alloys.
Chapter 2 : Study and improvement of the manufacturing process

This first study on the laser melting process using powder bed has been realised with the objective of identifying and avoiding the main defects caused by a bad control of the material's fusion. It was although interesting for the client company to analyse more precisely the defected parts and optimize the production settings like the laser’s parameters after a recent update of the equipment.

1 Equipment and parameters

The following study is about the Selective Laser Melting technology, it’s used here to manufacture Dental implants with the Cobalt Chromium Molybdenum alloy. Its technical characteristics and chemical composition are the following ones:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm³)</td>
<td>8-9</td>
</tr>
<tr>
<td>Tensile strength (Mpa)</td>
<td>520-825</td>
</tr>
<tr>
<td>Yield strength (MPa)</td>
<td>460-690</td>
</tr>
<tr>
<td>Elongation at break (%)</td>
<td>1.5-15</td>
</tr>
<tr>
<td>Young's Modulus (GPa)</td>
<td>145-230</td>
</tr>
<tr>
<td>Vickers Hardness (HV)</td>
<td>300-465</td>
</tr>
<tr>
<td>Thermal Expansion Coefficient (10^-6/°C)</td>
<td>13-15</td>
</tr>
<tr>
<td>Melting temperature (°C)</td>
<td>1250-1500</td>
</tr>
<tr>
<td>Casting temperature (°C)</td>
<td>1300-1600</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co</td>
<td>59%</td>
</tr>
<tr>
<td>Cr</td>
<td>25%</td>
</tr>
<tr>
<td>W</td>
<td>9.5%</td>
</tr>
<tr>
<td>Mo</td>
<td>3.5%</td>
</tr>
<tr>
<td>Si</td>
<td>1% max</td>
</tr>
<tr>
<td>Fe</td>
<td>1.5% max</td>
</tr>
<tr>
<td>N</td>
<td>1.5% max</td>
</tr>
<tr>
<td>C</td>
<td>1.5% max</td>
</tr>
<tr>
<td>Mn</td>
<td>1.5% max</td>
</tr>
</tbody>
</table>

The particles of the powder used in the process have an initial average diameter of 55 micrometres. When used at least once but not solidified in the manufactured parts, the powder is recycled after a mechanical screening at 80 micrometres. This screening limits the presence of particles with an unacceptable non spherical geometry that could cause manufacturing defects. Indeed, when not merged whit each other, the powder’s particles can still create material agglomerations.
An analysis of the new and recycled powders has been made with a Scanning Electron Microscope to check those dimensions and aspects. The new powder presents a normal distribution around the mean value measured here at 53 micrometres. In the recycled one, the distribution is basically the same for the majority of the particles but some have a larger diameter of 84 micrometres when resulting from an agglomeration. Some other particles are smaller when partially melted.

![Image of new and recycled powder](image)

*Figure 13: New (left) and recycled (right) powder*

The machine used for this study is the SLM100, produced by ReaLizer with a height of 2.1m, a width of 1m and a depth of 0.85m for a total mass of 500kg.

![Image of SLM 100 machine](image)

*Figure 14: SLM 100 (ReaLizer / Globatek.3D)*

The printing area's dimensions are 125*125*100mm (XYZ) and it’s possible to manufacture with layers from 20 to 100 micrometres. The laser is moved in the XY plane of the work area thanks to two mirrors and the vertical displacement of the manufactured part is linked to the support that move downward at each iteration of the printing process respecting the depth of the layers.
The two mirrors rotating around their own axis constitute the galvanometric head and make part of the optic mechanism of the machine. Before this step, the optic fibre encounters a collimator that creates a beam constituted of several parallel smaller beams. Then, this beam passes through a beam expender that increase its diameter. The last step is an F-Theta lens that focalize the laser on the printing area. Thus, the diameter of the laser and the energy transmitted to the material are equals on all this area. The laser used here is an Ytterbium Fibre type, YLR-200-WC, and is water-cooled. Its initial diameter is 370 micrometres and can be reduced to 20 thanks to the lenses system, its power goes from 20 to 200W and its wavelength is 1070nm.

The manufacturing is made under controlled atmosphere with Argon and 0.2% of Oxygen and a consumption of 35 litres per hour.

To control the process, two software developed by ReaLizer are necessary: Designer and Operator. The first one is used to prepare the manufacturing: defining the geometry of the part and the process parameters as the depth of the layers or the lasing strategy for example. Operator is used to automatically control the process and monitor in real time the state of the machine.

For this study, the first step has been the identification of the different manufacturing process' parameters:

- Laser parameters:
  - The focalization: before, on or after the last layer
  - The diameter
  - The power defined by the injection current's intensity
- The lasing strategy
  - In homogeneous layers crossed or not
  - The sweeping step
  - The speed
    - The exposure time of the powder to the laser
    - The point to point distance, distance between to following lasers’ impacts
The part orientation: inclined at 0 or 45° for example

- The layer depth

- The design of the supports to obtain a good quality of the first iterations supporting then the rest of the part

- The printing area temperature and the stress relieving heat post-treatment composed of a holding time of some hours at temperature followed by a slow cooling. Those two are used to reduce stresses in the manufacturing parts causing their deformation.

Two interactions are possible between:

- The beam size and the sweeping step giving the recovery rate between two successive laser passages

- The power and the speed of the laser that can give:
  - A poor fusion resulting in a poor density of the part if the laser goes too fast and its power is too low
  - Too big stresses in the parts causing cracks and elements' vaporisation is the laser goes too slow and its power is too high

A choice has been made to fix some parameters in order to focalize on the most influent and interesting ones here. The manufacturing process has been studied with two objectives:

- Identify the limits of the manufacturing process with the different defects that can occur in the parts at a macro or microscopic level at their surface or at their heart

- Find the configuration that gives the best visual quality
The fixed parameters in the study are the following ones:

- The diameter of the laser spot: 140µm
- The lasing strategy is crossed at 90°, the laser goes along the X axis on a layer and on the Y axis on the following one
- The sweeping step: 80µm
- The printing orientation: horizontal
- The layer depth: 50µm

Thus, no heat is applied to the printing area and no heat post-treatment is made.

The supports of the parts are automatically designed and optimized with Magics, a software from Materialise. Their manufacturing parameters are the following ones:

- Intensity of the laser’s injection current: 2000mA
- Focalisation of the laser on the layer
- Pdist: 20µm
- Texpo: 45µs

Thus, the most interesting parameters studied here are the following ones:

- The point to point distance
- The exposure time
- The laser power
2 Study of the manufacturing defects

2.1 Identification of the defects

A first experiment design has been made with the point to point distance and the exposure time defined on five levels with large range of values to explore the limits of the manufacturing process. The intensity of the laser’s injection current is fixed at 3600mA and the focalisation occur at 250 micrometres above the layer. 25 cubic parts of 10*10*5mm have been printed with those values:

- \( \text{Pdist}: 10, 40, 65, 90, 120 \mu\text{m} \)
- \( \text{Texpo}: 10, 40, 65, 90, 120 \mu\text{s} \)

A first visual analysis leads to a conclusion on what could be the most interesting values for the exposure time and the point to point distance. Four types of visual defects are identified:

- The supercooling
- The collapse of the upper face
- Cracks caused by the internal stresses
- The poor density of the part caused by the non-solidification of particles

The supercooling gives smoothness to the part but, on the other hand, causes its deformation that can oblige to stop the manufacturing if it’s too important. It is then necessary to reduce the exposition of the powder to the laser. The results showed, indeed, that an exposure time superior or equal to 90 microseconds with a point to point distance too small, like 10 micrometres, were causing supercooling.

The collapse of the upper face leads to a lack of fidelity of the part to the numerical model. The identification of this defect can be difficult, it can be compared to the supercooling one but with a less severe impact. However, as long as it’s necessary to respect the specified geometry and the flatness of the surfaces, it’s important to eliminate this defect. It can occur with different values of the parameters but is always linked to an unbalance between them. In fact, it appears when the exposure time is too high compared to the point to point distance, even if it is smaller than 120 microseconds.
The cracks are caused by too high internal stresses. It is known that the parts produced with additive manufacturing have an important internal stresses state because of the big variation of the powder’s temperature caused by the repeated scans of the laser. Like the precedent defect described, a balance has to be find to limit the occurrence of cracks. The observations made show that a point to point distance too high compared to the exposure time causes cracks, even if it is smaller than 120 micrometres.

The last identified defect is the bad or non-solidification of the powder. It is clear that such a defect appears when the exposition of the material to the laser is too low. The visual analysis confirm this: a too small exposure time, like 10 microseconds, with a point to point distance superior or equal to 90 micrometres doesn’t lead to a correct fusion of the powder’s particles between each other.

First of all, to avoid parts with those defects, it’s necessary to reduce the range of values of those two parameters. By eliminating the extremes, the exposure time is limited between 40 and 90 microseconds and the point to point distance between 40 and 90 micrometres.

By working at a constant laser’s power, a good balance between point to point distance and exposure time leads to a good visual quality to the parts and:

- The superiority of the point to point distance causes cracks and then poor density when the gap increases
- The superiority of the exposure time causes the collapse of the upper face and then supercooling when the gap increases

Figure 16: Influence of the point to point distance and the exposure time on the visual defects observed
2.2 Analysis of the defects

Four defected samples and a correct one have then been chosen to realize studies of surface state’s characterization and electron and optical microscopy analysis:

<table>
<thead>
<tr>
<th>Sample</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pdist (µm)</td>
<td>10</td>
<td>10</td>
<td>40</td>
<td>120</td>
<td>120</td>
</tr>
<tr>
<td>Texpo (µs)</td>
<td>10</td>
<td>90</td>
<td>65</td>
<td>90</td>
<td>40</td>
</tr>
<tr>
<td>State</td>
<td>Correct</td>
<td>Surpercooled</td>
<td>Collapsed</td>
<td>Cracks</td>
<td>Cracks, Poor solidification</td>
</tr>
</tbody>
</table>

The fourth and last defect described that is the non-solidified state of the part is impossible to analyse because it doesn’t lead to exploitable parts. However, the fifth sample presents cracks but is close to the non-solidification defect by its manufacturing parameters’ values.

2.2.1 Study of the roughness

The surface state’s measures have been realised on profile and 1mm² squared surfaces, leading too to topography analysis, thanks to the topographic measurement station Altisurf (Appendix 1).

The roughness is evaluated with the criteria Ra or arithmetical average gap of the profile on an average length of 10mm. The criteria Sa is although used, it is similar to Ra but extended to a surface, the arithmetical average height.

The topographic analysis of the squared surface allows to realize its mapping and, then, to identify the peaks and valleys: it allows to visualize the global state of homogeneity of the surface.

<table>
<thead>
<tr>
<th>Sample</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra (µm)</td>
<td>19</td>
<td>4.63</td>
<td>15.3</td>
<td>12.7</td>
<td>37.6</td>
</tr>
<tr>
<td>Sa (µm)</td>
<td>16.9</td>
<td>5.9</td>
<td>17.6</td>
<td>15.9</td>
<td>45.4</td>
</tr>
<tr>
<td>Peak to valley distance (µm)</td>
<td>65</td>
<td>80</td>
<td>170</td>
<td>240</td>
<td>350</td>
</tr>
</tbody>
</table>
Two different evolutions of the roughness with the manufacturing parameters can be identified:

- With a constant point to point distance, the roughness increases with the decrease of the exposure time
- With a constant exposure time, the roughness increases with the point to point distance

In fact, decreasing the exposure time of the powder to the laser is like decreasing the quantity of melted material and, then, increases the roughness because of the non-homogeneity of the surface. The increase of the point to point distance leads to the same effects because of the same reasons.
The mapping leads to a first evaluation of the surfaces’ collapse. For example, the sample 1 presents a really good homogeneity and really little collapse of the surface. In comparison, the sample 2 is a little less homogeneous and presents more collapse of its upper surface but its analysis allows to verify its low roughness previously observed.

The samples 3 and 4, too, presents good homogeneity but the collapse of their surfaces is more important with a punctual defect for the sample 3 and bigger global collapse for the sample 4.

The sample 5, compared to all the others is really inhomogeneous and presents a big collapse in localized areas of its surface as it was predictable with its high roughness. It can be explained with the low cohesion of the powder’s particles for this sample presenting cracks and being at the limit of the non-solidification. Those particles don’t have the time to fuse to create a layer with a good homogeneity and, then, the surface has a lot of asperities and holes.

It’s difficult to conclude on the quality of the parts with the only analysis of their roughness. It’s necessary to cross these results with the macro or microscopic visual analysis.

In fact, for the sample with supercooling defect, the roughness let think about an excellent surface’s state. The surface is really smooth but it’s due to the too big intakes of energy that receive the powder with the laser layer after layer. An additional analysis has to be made at the heart of the part to see the material’s cohesion’s state.

Similarly, the sample whose state is correct has a roughness in the same range of the samples with big internal stresses or collapse, though it’s more homogenous and less collapsed. An additional analysis is necessary too at the heart of the part to verify the good state of the part.

2.2.2 Microscopic study

This study has been realised with an Inversed Optical Microscope (Appendix 2) and a Scanning Electron Microscope (SEM: Appendix 3). The analysis is done at the surface and at the heart of the parts to visualize porosities, cracks and the imprints let by the laser on the powder during the manufacturing process.
To proceed to this study, the samples have been prepared with the method explained in the Appendix 4. They have been cut, enveloped and then mirror-polished. Then, an etching is necessary to reveal the material microstructure. According to the ASM Handbook Metallography and Microstructures, it has been decided to realise an electrochemical one during 15 seconds at 3 volts in a solution composed with 50% of nitric acid and 50% of water.

The analysis at the surface of the sample 1 allowed to identify the imprints of the laser, the non-melted powder’s particles and some porosities in low quantity. It is, then, possible to measure the width of the imprints as the width of the laser’s thermal effect. The manufacturing’s good quality can be confirmed as long as this measure matches with the sweeping step of 80 micrometres. The measure of a particle’s diameter confirms too the average size of the new powder of 55 micrometres.

![Image of sample analysis](image)

*Figure 18: Surface (up) and sectional view (down) of the sample 1*

The analysis of the cut shows some porosities, around 20 micrometres, which the quantity can be evaluated with an images analysis when the sample is mirror-polished. The imprints
of the laser can be seen too with the scanning strategy, layer by layer and crossed at 90°. This imprints have the shape of weld seams in the form of arcs of circles when seen by profile. The radius of these arcs gives an information about the depth of powder affected by the laser. The measure of this depth shows a process well optimized in this case because it is more or less equal to the dimension of the layers of 50 micrometres.

The sample 2 has some porosities at its surface too and non-melted particles. The measure of their size gives the same result as the sample 1. However, the imprints of the laser are more evident than for the sample 1. Those weld seams are more inhomogeneous: their central area that received more energy is smooth when their edges present accumulations of material. In this case, because of the overexposure of the powder to the laser and of the diffusion of the heat, the width of the seams is almost two times larger than the sweeping step. For the sample 1, the seams were better defined and the energy transmitted to the material lower: this allows a better control of the powder's fusion and cooling and of the manufacturing process in general.

![Figure 19: Surface (up) and sectional view (down) of the sample 2](image)
The cut of this sample reveals a lot of porosities and a crack that were not visible at the surface. As it was predictable, the supercooling gives a good and smooth surface’s state with low roughness which is way different that the heart’s state. The measure of the depth of the seams which such a high exposure of the powder to the laser gives a value a lot bigger than the one used for the layers.

The collapsed surface of the sample 3 shows some powder’s particles too and the width of the weld seams is measured more or less twice bigger than the sweeping step used because of the too high exposure of the powder. It’s although possible to analyse the imprint of the laser and the strategy used on the borders of the sample’s surface.

![Image](image.png)

*Figure 20: Surface (up) and sectional view (down) of the sample 3*

The cut’s study reveals a correct amount of porosities closed to the one measured with the sample 1 when the depth of the seams is a bit bigger than the layers’ one.
The sample 4 presents an internal stresses’ state too important but it can’t be seen at its surface. The width of the weld seams is close to the sweeping step and some non-melted powder’s particles are present.

On the contrary, the cut’s study reveals porosities with an important size and cracks due to the presence of internal stresses. When the surface analyse showed a sample’s state close to the first one, the cut reveals the difference. Another particularity of this sample is the depth of the weld seams bigger than in the case of the sample 1. The laser melts the last layer but with this exposure can although bring back in fusion until two already solidified layers. The optimization of the laser’s parameters is done to avoid this effect because those repeated supplies of energy to the powder lead to the inhomogeneity of the material thermal’s history. It’s this inhomogeneity that give the important internal stresses’ state and the macroscopic cracks.

The sample 5 presents cracks like the precedent but poor solidification too because of its lower exposure time. At its surface, a lot more porosities and non-melted particles can be seen compared to the sample 4. Their size goes from 55 micrometres, the new powder’s
diameter, to 80 micrometres, the recycled one’s after the screening. It confirms the defect observed on this sample that is the poor density and solidification of the material. The visualisation of the weld seams is difficult due to this high manufacturing’s inhomogeneity.

![Sample analysis images](image)

**Figure 22: Surface (up) and sectional view (down) of the sample 5**

The cut’s analysis of this shows the important inhomogeneity too with a lot of porosities and cracks. It’s possible to analyse some weld seams’ depth and the measure reveals a value smaller than the layer’s depth. Thus, the poor solidification and general bad finite state can be explained: the measure of the porosities’ quantity reveals that the material is only melted at 50%.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weld seams</th>
<th>State - Pdist / Texpo</th>
<th>Porosities (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>84.8</td>
<td>50.8</td>
<td>Correct - 10 / 10</td>
</tr>
<tr>
<td>2</td>
<td>136.3</td>
<td>153.3</td>
<td>Supercooled - 10 / 90</td>
</tr>
<tr>
<td>3</td>
<td>180.3</td>
<td>64.4</td>
<td>Collapsed - 40 / 65</td>
</tr>
<tr>
<td>4</td>
<td>80</td>
<td>66.5</td>
<td>Cracks - 120 / 90</td>
</tr>
<tr>
<td>5</td>
<td>Unmeasurable</td>
<td>32.8</td>
<td>Cracks, Poor solidification - 120 / 40</td>
</tr>
</tbody>
</table>
To conclude this microscopic study, the sample 1 confirms at heart the correct macroscopic state at surface. The laser’s parameters here allow to respect the manufacturing’s ones like the layer depth and the sweeping step with a controlled fusion. Thus, the amount of powder put in fusion by the laser is optimized and the part presents a good roughness and low amount of porosities.

The supercooled sample seemed to be interesting at the surface with the lowest roughness but, as predictable, the study at heart showed big porosities and revealed a thermal influence’s area of the laser too important. The uncontrolled fusion due to the bigger exposure leads to an inhomogeneous part.

The poor solidified sample, compared with the supercooled one, was clearly identified as defective with its high roughness at the surface and the too low exposure to the laser’s energy leading to its poor density and a really important amount of porosities.

The collapsed sample and the one presenting cracks had not a lot of porosities and a correct roughness but the fusion is not perfectly controlled and optimized in those cases. Those two defects are more difficult to identify comparing with the two others which are extremes cases of bad manufacturing. They are really close to be considered as non-defective parts because the balance between the exposure time and the point to point distance is almost respected as it’s been showed earlier. Only a complete analysis at heart as at the surface can lead to a good differentiation and control of the manufacturing process.
3 Study of optimization of the parts’ visual aspects: roughness and collapse effect

3.1 Exposure time and point to point distance

A new experiment design has been realised in order to improve the visual aspect of the parts optimizing the manufacturing parameters. The two main studied characteristics to validate the results are the roughness and the collapse effect. It seems necessary to respect the numerical model during the manufacturing process as long as here for a biomedical application with the production of dental prostheses. Those prosthesis are then enveloped and don’t have to resist to big efforts. That’s why, in the present case, the internal and mechanical aspects of the produced parts are less important.

The starting point of this study is a set of parameters already identified by the manufacturer as interesting before an upgrading of the machine. The parameters are the following ones:

- Focalisation: 180 micrometres above the layer of powder
- Intensity of the laser’s injection current: 3600mA
- Point to point distance: 40µm
- Exposure time: 60µs

The roughness of this reference sample has been measured:

<table>
<thead>
<tr>
<th>Ra (µm)</th>
<th>Sa (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18,2</td>
<td>22,9</td>
</tr>
</tbody>
</table>
The roughness of this sample is certainly higher of the ones measured in the previous study but the samples had been identified as defective like the one supercooled.

At first, it has been decided to analyse small variations of the exposure time and the point to point distance around the reference values with 42 cubic samples of 5*5*5mm:

- Texpo, 7 levels between 30 and 90µs: 30, 40, 50, 60, 70, 80, 90
- Pdist, 6 levels between 20 and 70µm: 20, 30, 40, 50, 60, 70
After a first visual analysis, it is possible to identify an area with supercooling effect where the part’s supports are damaged and the upper face is collapsed and smooth. This area is delimited with a small point to point distance and a big exposure time.

Another identifiable area is the one with small exposure time and a big point to point distance. In this one the roughness is high but the upper face is plane. It although looks like the solidification of the samples in this area is bad.

The roughness of the parts manufactured with small values of the exposure time and point to point distance seems to be correct but the collapse of the upper face in this case is important.

The area that seems to be the most interesting one here is the one using high values for the exposure time and point to point distance. The roughness is similar than the one is the precedent defined area but the collapse effect less important.

For this area and more precisely for the following sample, the topography and the roughness has been measured with a surface and a profile:

<table>
<thead>
<tr>
<th>Pdist (µm)</th>
<th>Texpo (µs)</th>
<th>Sample</th>
<th>Reference</th>
<th>Optimisation 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>70</td>
<td>90</td>
<td>Ra (µm)</td>
<td>18,2</td>
<td>21,3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sa (µm)</td>
<td>22,9</td>
<td>29,4</td>
</tr>
</tbody>
</table>
The visual aspect of these samples is correct but the roughness' study showed that is increased compared to the reference sample. Indeed, the most interesting parameters seem to be between this area and the reference sample and these are the ones that will be used for the rest of the study with the laser's power.

### 3.2 Laser’s power

This new experiment design allowed the study of the influence of the laser's power. The parameters of point to point distance and exposure time have been chosen and fixed close to the reference ones as they have been defined as leading to satisfying results in the precedent study. The sets chosen are the following ones:

<table>
<thead>
<tr>
<th>Pdist (µm)</th>
<th>40</th>
<th>40</th>
<th>50</th>
<th>50</th>
<th>60</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>Texpo (µs)</td>
<td>60</td>
<td>70</td>
<td>60</td>
<td>70</td>
<td>60</td>
<td>70</td>
</tr>
</tbody>
</table>

The laser power is studied on 4 levels with the intensity of its injection current: 1.2A, 2.4, 3.6A and 4.8A: it leads to 24 samples of 5*5*5mm.
A first visual analysis leads to conclusion that the increase of the laser’s power make decrease the roughness but make increase the collapse effect of the part. A balance has to be find between a good surface’s state and the respect of the numerical model's geometry. Another thing is that at constant power, increasing the point to point distance increases the roughness.

Thus, the most interesting parts when studying the roughness are the ones manufactured with a high laser’s power and a small point to point distance, here 40 micrometres. Indeed, the best result, putting apart the collapse effect, is the reference sample with an increase of the laser’s power, here with 4.8A for the intensity of its injection current.

Analysis of the most interesting sample’s roughness:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Reference</th>
<th>Optimisation 1</th>
<th>Optimisation 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra (μm)</td>
<td>18.2</td>
<td>21.3</td>
<td>14.3</td>
</tr>
<tr>
<td>Sa (μm)</td>
<td>22.9</td>
<td>29.4</td>
<td>22.2</td>
</tr>
</tbody>
</table>
The roughness’ study of this sample confirms the visual result when it’s compared with the reference sample. At a constant point to point distance and exposure time, increasing the laser’s power allows to reduce the roughness.

This experiment design although allows to see that a too low intensity of the injection current, like 1.2 or 2.4A doesn’t lead to a good solidification of the material. It’s even more visible with the connection between the supports and the parts. The supports are manufactured with an intensity of 2A to be easily eliminated but this value is too low to be used for the full parts. In those cases the intensities used are too close to this value and even inferior what causes the defect with first layers manufactured.

As long as the increase of the power reduce the roughness but leads to the collapse effect, it has been decided to optimize the power focusing on samples with a bad roughness but a good respect of the numerical model. Six set of parameters have been chosen in the previous experiment designs:

<table>
<thead>
<tr>
<th>Pdist (µm)</th>
<th>60</th>
<th>60</th>
<th>60</th>
<th>70</th>
<th>70</th>
<th>70</th>
</tr>
</thead>
<tbody>
<tr>
<td>Texpo (µs)</td>
<td>30</td>
<td>40</td>
<td>50</td>
<td>30</td>
<td>40</td>
<td>50</td>
</tr>
</tbody>
</table>
The interest of this experiment design is that it could be possible to improve the roughness by increasing the power and to keep at the same time the quality of the initial sample with its good flatness.

30 samples have been produced with the laser’s power studied on 5 levels with its injection current’s intensity: 3.6A, 4A, 4.4A, 4.8A, 5A.

![Experiment design 3](image)

The visual analysis of the samples leads to focus on an area where the set of parameters give a good balance between roughness and collapse effect.

Then, the most interesting sample is the one manufactured with a point to point distance of 60 micrometres and an exposure time of 50 microseconds with an intensity of 5A. Nevertheless, it seems that such a high intensity leads to defects at the junction with the supports. The precedent design showed that the intensity can’t be too low and this one put a maximum to it. In fact, the sample produced with the same time and distance but an intensity of 4.8A presents a good roughness and low collapse effect too and avoids this defect at the lower surface. A small decrease of the laser’s power allows a better control of the powder’s fusion and thus a better respect of the numerical model.

Analysis of the most interesting sample’s roughness:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Reference</th>
<th>Optimisation 1</th>
<th>Optimisation 2</th>
<th>Optimisation 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra (μm)</td>
<td>18.2</td>
<td>21.3</td>
<td>14.3</td>
<td>13.1</td>
</tr>
<tr>
<td>Sa (μm)</td>
<td>22.9</td>
<td>29.4</td>
<td>22.2</td>
<td>14</td>
</tr>
</tbody>
</table>
Another time the roughness’ measure validated the quality’s improvement of the sample comparing with the precedent design. The roughness measured here with the final sample is the best result among the different steps of optimizations. Increasing the laser’s power allowed to eliminate the parts’ defects without decreasing their quality.

To conclude this study of the parts’ aspect’s optimization, the manufacturing parameters deducted from the different experiment designs are:

- Pdist: 60µm
- Texpo: 50µs
- Injection current’s intensity: between 4.8 and 5A

The study has been realised on the laser’s power and speed parameters because they had been defined as the most influencing ones on the production’s quality. However, the final optimized sample has other parameters, fixed for the present study, which could be part of an additional study to a better improvement of the parts’ aspect.
These interesting parameters are the following ones:

- Focalization of the laser: at 180 micrometres above the powder’s layer
- Diameter of the laser’s spot: 140µm
- Sweeping step: 80µm
- Printing direction: horizontal
- Layer depth: 50µm

To conclude this manufacturing process study with 3D Dental Store, it’s been possible to bring them concrete analyses results with the microscopic identification of defected parts when they don’t seem to have conception problems with a macroscopic observation. The different possible measures made on the laser’s influence zones allow to control the material fusion’s quality and porosities are observable at heart. It’s been finally concluded an optimized set of laser’s parameters for the precise application of this dental manufacturer looking for the improvement of the parts’ surface state.
Chapter 3 : Topological optimization study

1 Introduction

The topological optimization is perfectly adapted in the reconception process for additive manufacturing. It can be applied on structure parts of light electric vehicles to improve their mobility by reducing their mass. The present study focused on the suspension system of the small two-seat vehicle from Renault, the Twizy.

The part that can be interesting to optimize is the suspension’s triangle: this one is linked to the chassis by two silentblocs and to the hub carrier by two kneecap connections. It’s this hub carrier that support the brake calliper and the damper and spring system.
This stainless steel part although presents two holes at its centre to relieve the stresses concentration, these holes are important to improve the resistance during the use, for example with a tension-compression effort. During the optimization study those holes won’t be necessary as long as the change of the part’s geometry can change the areas where the efforts transit.

This suspension system linking the ground to the vehicle is a security part and thus has to resist to static and dynamic efforts, to vibratory stresses and impacts. These impacts can occur against the pavement for example and are transversal compressing the triangle or longitudinal torsioning it. Another effort is the vertical one flexing the part but this vertical stress is lower thanks to the damper and spring system on a side and the silentblocs on the other both absorbing energy and reducing the stress.

The optimization is realised with the objective of a metal additive manufacturing with the Direct Metal Laser Sintering process using a powder of stainless steel 316L.

To realise the optimization, a software of 3D numerical modelling, finite elements analysis and topological calculation have been used. First, it’s been necessary to realize the functional analysis of the original part with ANSYS. But to get mechanical information about the additive manufactured 316L, tensile tests have although been made. When known the limit loads that can support the part in flexion, tension and compression, these parameters are entered in the topological optimization software Inspire from solidThinking or HyperWorks from Altair. The calculation gives an idea optimized part's geometry when focusing on its stiffness or its mass. Then, the part is redesigned on SolidWorks and resimulate on ANSYS to validate its optimization.
2 Tensile-tests: anisotropy’s study and analysis of the break

2.1 Mechanical characteristics measures

To realise the topological optimization study, as every numerical simulation, it’s necessary to well define the entry parameters and the test’s specification: materials, supports and efforts. The software already have predefined parameters for most of the conventional materials but the data doesn’t correspond with additive manufactured parts. It’s then necessary to make mechanical tests to obtain and analyse the different characteristics that a literature review or a powder’s and machines’ producer wouldn’t give as precisely. In fact, the process’ repeatability may not be optimal even if the manufacturing parameters are sold by the producer as an optimized set.

The anisotropy’s study is although really important in the Metal Additive Manufacturing processes. It’s necessary to quantify the tensile behaviour depending on the printing strategy to guess the best manufacturing solution according to the specific use of the part. To test the influence of the manufacturing direction on the parts resistance and obtain the principals mechanical characteristics of the additive manufactured 316L, tensile tests have been realised. The specimens are flat and printed in two different ways:

- 3 “horizontal” specimens, made by stacking the layers parallel to the tension direction, in the thickness’ direction. The solicitation is longitudinal in the direction of the layers
- 3 “vertical” specimens, made by stacking the layers perpendicularly to the tension direction, in the length’s direction. In this case the solicitation is transversal.

![Figure 32: Directions of the tensions](image)
These specimens are heat-treated after the manufacturing process to relieve the stresses before the separation from the support by Electrical Discharge Machining and a shot peening of the surfaces (Appendixes 5 and 6). The heat treatment avoids the part's deformation, it's heated at 650°C during 2h and then slowly cooled in the furnace.

The manufacturing layer by layer necessarily implies a weakness in the parts in the bounds between the layers. It can be predicted that a tension realised parallel to the manufacturing direction will produce more negative effects on the material cohesion than the one realised perpendicularly: it's this anisotropy that will be quantified here.

The tensile tests realised at the mechanicals tests laboratory LHEM allowed to measure the following characteristics:

- Tensile strength, $R_m$ in MPa
- Yield strength, $R_p$ in MPa
- Elongation at break, $A$ in %
- The maximum force at rupture, $F_m$ in N
- The Young’s modulus, $E$ in GPa

The method used to measure the yield strength is the conventional one, $R_p0.2$ leading to the stress giving 0.2% of residual deformation. This method can be used as long as the material is ductile and goes smoothly from an elastic to a plastic behaviour.
The results are the following:

<table>
<thead>
<tr>
<th></th>
<th>Horizontal</th>
<th>Vertical</th>
<th>Anisotropy</th>
<th>Conventional</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rm (MPa)</td>
<td>632 ± 4,2</td>
<td>521 ± 1,2</td>
<td>21%</td>
<td>400-860</td>
</tr>
<tr>
<td>Rp0,2% (MPa)</td>
<td>463 ± 5,2</td>
<td>398 ± 32</td>
<td>16%</td>
<td>180-350</td>
</tr>
<tr>
<td>A%</td>
<td>29 ± 0,6</td>
<td>40 ± 3,4</td>
<td>-28%</td>
<td>20-60</td>
</tr>
<tr>
<td>Fm (N)</td>
<td>19829 ± 158</td>
<td>16632 ± 52</td>
<td>19%</td>
<td></td>
</tr>
<tr>
<td>Young's modulus (GPa)</td>
<td>168 ± 35</td>
<td>136 ± 16</td>
<td>24%</td>
<td>185-210</td>
</tr>
</tbody>
</table>

The numerical results show an evident anisotropy between the two manufacturing strategies with an average difference of 20%. The horizontal specimens have a better resistance to the tension with bigger maximal supported efforts and a higher yield strength when the vertical ones have better ductility. These vertical specimens allow a bigger elongation at break but with a lower maximum force.

The Young's modulus is although higher for the horizontal specimens validating its better resistance to the tension. However this measure shows an important dispersion until 10% for the horizontal specimens but can still confirm the average anisotropy of 20% observed with the other characteristics.

![Young's modulus graph](image)

Other measures showed a significant dispersion like the elongation at break or the yield strength for the vertical specimens: this is interpreted as a repeatability defect of the manufacturing process. The tensile tests require a good manufacturing precision as long as the specimens’ geometry is defined with a tolerance by norms, here the NFEN ISO 6892-1 B. This norm implies a machining tolerance of 12.5±0,05mm on the useful zone’s width and
allows then a test’s speed based on the loading speed of 10N/mm²/s. This level of precision is not possible with the Metal Additive Manufacturing and this width had to be oversized and machined to respect the norm.

It’s although possible to see precision’s errors at the level of the specimens’ depth. In fact the theoretical depth is 2.5mm but for both of the specimens’ types the measures value is bigger. The vertical strategy is the more unprecise with a depth of 2.563mm, a difference of 2.52%. In comparison the horizontal strategy leads to a depth of 2.503mm, a difference of 0.12%.

A literature review of these same characteristics for a 316L stainless steel conventionally produced showed similar elongation at break and tensile strength. However the yield strength is two times smaller when the part is not additive manufactured: this can be explain with the lower cohesion of the material between the layers with an additive manufacturing process. Then, the part can suffer a bigger elongation before a break or a plastic deformation. The Young’s modulus is although smaller for the specimen issued from the additive manufacturing process.

On the tensile-test curves, the two types of solicitations appear to have a ductile response. However, the horizontal specimens are seen as having a better resistance to the tension with a bigger maximum of tensile stress even if its break is premature.

This better resistance is explained by a better cohesion of the material for the horizontal manufacturing strategy. In fact, the stress applies on all the length of the layers stretching them without degrading their cohesion. On the contrary, with the vertical specimens, the
layers are stressed in their depth and are going to be teared apart. Then, the part loses its cohesion at the layers joints.

After reaching its maximum, the tensile stress decreases with the increase of the elongation because of the necking effect. The necking is a transversal shrinkage: the section of the specimen decreases while its length increases. The necking is more important in the case of the vertical specimens as long as they resist more before breaking by elongating deformation.

Linked with this better capacity to the deformation, the plastic deformation zone is more important in the vertical specimens' case when the horizontal ones have a better elasticity. The lack of cohesion of the vertical specimens allows in return a better capacity to the rearrangement of the material before the break.

2.2 Tensile-test analysis before and after the break

Various analysis have been realised on the specimens before and after the tensile tests. Before the test, the roughness has been measured with profiles of a length of 12.5mm in the width and of 100mm in the length. It has although been measured with squared surfaces of 1mm². It showed a roughness a bit more important for the horizontal specimens knowing that all the surfaces have been shot peened. The measured surface for the horizontal specimens is the first or last fused layer when in the case of the vertical specimens, it’s composed of following layers seen by profile. Then, the measured surface potentially presents more asperities in the case of the horizontal specimens with the laser melting process’ marks.

<table>
<thead>
<tr>
<th>Tensile-test specimen</th>
<th>Ra (μm) length</th>
<th>Ra (μm) width</th>
<th>Sa (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vertical</td>
<td>7.9</td>
<td>7.2</td>
<td>7.7</td>
</tr>
<tr>
<td>Horizontal</td>
<td>12.2</td>
<td>10</td>
<td>11.2</td>
</tr>
</tbody>
</table>

After the test, the specimens have been analysed with optical and electron microscopes to make a fractography. The hardness has although been measured at the extremities of the specimens and close to break area to see its evolution with the deformation.

2.2.1 Break analysis

With a material like the stainless steel 316L, the expected type of break is ductile with necking. It should be possible to observe craters or cupules with inclusions in a plan of break
oriented at 45°. This ductile behaviour is predictable with the important elongation measured before, around 30-40%. For a ductile break it’s although common to observe transgranular breaks with plastic deformed zones giving a matte and rough aspect to the surface. There should not be preferential initiation zones or propagation directions: this steel having a good formability, will resist to the cracks’ propagation with a deformation under stress.

To control those characteristics in the case of a specimen additive manufactured, a first macroscopic observations is done and various types of break are identified.

![Figure 34: Tensile-test specimens after the break, Vertical (up) and Horizontal (down)](image)

The different specimens have a matte break surface as predicted what confirm the ductile break. However the break is different depending the vertical or horizontal manufacturing strategy. The vertical specimens' breaks are regular and clean with this specific orientation at 45° but the horizontal specimens' break seems to be more aleatory. It initiates sometimes the same way as the vertical ones, sometimes from the both sides of the specimen and sometimes in a symmetrical way from the centre of the section.

![Figure 35: Vertical tensile-test specimen (left) and Horizontal (right)](image)

All the breaking surfaces have dark spots appeared during the tensile test. They correspond to fragilities zones, internal stresses and heating of the material issued from the test.
Microscopic pictures allowed to see the deformation of the break section, the vertical specimens show more deformation as resulting from a torsion stress when the horizontal ones have a straighter break. This confirms the necking observed before and depending on the vertical or horizontal strategy: indeed, this necking is more important for the vertical specimens.

![Figure 36: Vertical tensile-test specimen (left) and Horizontal (right), Break surface](image)

The analysis with the electron microscope allowed to make a more precise study of the break area with its surface state. It revealed cupules as it was expected for a ductile fracture and some smoother areas proof of the presence of fragilities. Thus, the global ductile behaviour macroscopically observed is confirmed.

![Figure 37: Vertical tensile-test specimen (left) and Horizontal (right) Break surface x2000](image)
2.2.2 Analysis of the break in the cut

The cut’s study has been realised with the optical and electron microscopes after the samples’ preparation by cutting, enveloping and polishing as explained in the Appendix 4. The etching used to reveal the material microstructure is the following one:

- Electrolytic etching at 2.5V during 15 seconds in a solution composed of 10% in mass of oxalic acid diluted in water.

At first, the microscopic analysis allows to see the weld seams issued from the laser melting process of the powder. They have the shape of superposed circles arcs when seen by profile and ellipses when seen in the plane. This superposition reveals the printing direction with the layers’ stacking process.

![Figure 38: Weld seams seen in the cut (left) and at the surface (right)](image)

It’s then possible to verify the lasing strategy used measuring the angle between the weld seams from a layer to another. The value of 67° is found that is a known strategy theoretically used to help to reduce the mechanical anisotropy.

The microscopic study in the cut of the horizontal specimens doesn’t show repeatability in the breaking process following the weld seams. On the contrary, it seems that the breaks occur at the heart of the melting pools more than at the borders. It will be seen in the following study that these central zones are the hardest and most fragile ones because of a faster cooling process.
In the case of the vertical specimens, on the contrary, the breaks follow more clearly the borders of the weld seams initiating between two layers. In fact, the tensile test makes the cohesion of the material decrease and, thus, the crack can propagate more easily between the layers. In the case of the vertical specimens, the layers are superposed in the same direction of the tensile test and perpendicularly to the preferential direction of the crack propagation. In the case of the horizontal specimens, the propagation of the crack is more difficult as well as the tearing apart of the layers. The crack during its propagation encounters perpendicularly the layers which, with their good cohesion, slow down its advance and improve its resistance to a higher tension force.

It's although possible to observe the elongation and the preferential orientation of the weld seams in the tension direction. Close to the break surface, the width and length of the seams increase for the horizontal specimens when it’s their depth for the vertical ones. These vertical samples although seem to have a cleaner break when analysing the enveloped break surface.
The cut’s analysis with the electron microscope allowed to see the porosities close to the surface break with more contrast. These ones are more important in the case of the vertical specimens because of the bigger elongation that they support. In fact, the porosities are revealed by the tensile test by the stretching of the material and induce fragilities.

![Porosities close to the break surface for the vertical specimens (left) and the horizontal ones (right)](image)

*Figure 41: Porosities close to the break surface for the vertical specimens (left) and the horizontal ones (right)*

The anisotropy observed with the mechanical characteristics and the tensions curves can be linked here with the one observed microscopically. The manufacturing strategy influences their microstructure with the weld seams orientation. The manufacturing by superposition of these seams implies weaknesses at their borders that lead to the formation of material’s sliding plans during the tension test.

In the case of the vertical specimens, more plasticity is observed with the necking effect and there are two types of seams’ borders. As show the next figure, are solicited the borders between two weld seams of two successive layers, layer-layer, and the ones between two weld seams of a same layer but of two successive laser’s steps, track-track. Only the track-track borders influence the break at the tension of the horizontal specimens. Thus, the vertical specimens present more sliding planes enabling the tearing apart of the layers with a better distribution of the stress between them and a more important final deformation. This better rearrangement of the material during the test lead to a bigger necking and elongation.

The horizontal printing strategy limits this deformation and thus the track-track planes, perpendicular to the tension direction, will suffer bigger stresses to finally break prematurely. These specimens are more fragile transmitting more effort before the break.
2.2.3 Hardness measure

For both types of specimens, a Vickers microhardness’ study has been realised at heart of the weld seams in cuts close to the break area and at the extremities of the specimens.

The measures follow the Vickers type HV0.1 method with a load of 0.1kg or 980.7mN during 10 seconds.

For the 316L stainless steel, a literature review showed a hardness around 120-140HV for a part issued from a conventional manufacturing process.

The value measured for both of the types of specimens and at their extremities is close to 220HV. No anisotropy is detected here but the hardness is a lot superior to the one issued from the literature review.

The measure close to the break surface is lot more interesting showing an anisotropy between the two manufacturing strategies and a big increase of the hardness after the tensile test. It’s the deformation of the material that lead to this increase of the hardness. The tension applied to the specimens makes appear internal stresses that had been partially eliminated with the stress relieving heat treatment.

These stresses induce dislocations, more important with the vertical specimens with the necking effect and this is confirmed with the hardness’ comparison. The vertical specimens presenting more deformation present although a higher hardness: the tensile test clearly reveal the anisotropy implied with the manufacturing strategy, here with a difference of 8% on this mechanical characteristic depending on the printing direction.
The indentations’ imprints present some deformations because of these internal stresses, the diagonals are measured between 27 and 28 micrometres at the extremities and around 21 micrometres close to the break area.

Figure 43: Indentations on the horizontal tensile-test specimens (up) and on the vertical ones (down) at the extremities (left) and in the break area (right)
3 Topological optimization calculation

The 316L stainless steel characteristics used for the calculation are the following ones issued from the tensile tests:

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm³)</th>
<th>Tensile strength (MPa)</th>
<th>Yield strength (MPa)</th>
<th>Young's modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L FAM</td>
<td>7.5</td>
<td>632</td>
<td>463</td>
<td>168</td>
</tr>
<tr>
<td>316L Conventional</td>
<td>7.9-8</td>
<td>400-860</td>
<td>180-350</td>
<td>185-210</td>
</tr>
</tbody>
</table>

A measure of the density has although been realised with a weighing and a volumetric measuring. It gives an order of magnitude of this characteristic for this part issued from an additive manufacturing process. As long as the manufacturing process is not a conventional machining parting from a raw material block or a casting, porosities or other geometrical irregularities can lead to a lower density as the one obtained conventionally: that’s the case here.

The first step consisted in the definition of the maximum static efforts that can stand the suspension’s triangle with the Von Mises equivalent stresses’ study. This is realised on ANSYS with the original part in 316L stainless steel fixed with the two silentblocs and submitted to three efforts on the kneecap connection. These efforts leading to the torsion, flexion and compression of the triangle are calculated in order to induce its deformation at the limit of its material’s elasticity when isolated or cumulated.

The part, weighting 1.36kg reaches 98.9% of its yield strength when submitted to the following efforts:

- Compression: 1750N
- Flexion: 205N
- Torsion: 6370N
The topological optimization study has been realised on two software produced by Altair. Inspire solidThinking allows a more educational approach with some simplified steps of the implementation when HyperWorks let more liberty in the choice of the study's parameters and thus allows a better calculation’s precisions. The results issued from this second software are exposed here.

The topological optimization is generally realised with two distinct objectives each one having its parameters and specific constraints:

- The mass minimizing using as criterion a security coefficient limiting the maximum of the average Von Mises stresses by defining it as a percentage of the yield strength.
- The stiffness maximizing using as criterion the retained part of the initial part’s volume: thus, the optimized part’s volume is defined as a percentage of the initial conception’s volume.

Here two mass minimizing studies have been tested with 1.5 and 2 as security coefficients, or 67 and 50% of the material yield strength and one stiffness maximizing one using 30% of the initial conception’s volume.

The initial implementation is realised with HyperMesh with the definition of the mesh, the efforts, the supports, the material characteristics and the part’s manufacturing symmetry. Then, each study has its parameters and constraints depending on its optimization objective as previously defined.

The solver used is OptiStruct and it, then, allows to visualise the results with HyperView as Von Mises stresses but although as an elements’ density mode. This mode gives visual
information about the relative importance of the different elements of the part, from 0 to 1, respecting the defined objective.

A first study is realised by default without focusing on the optimization specific parameters:

<table>
<thead>
<tr>
<th>Study</th>
<th>Maximise the stiffness - 30%</th>
<th>Minimize the mass – 1.5</th>
<th>Minimize the mass - 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Yield strength</td>
<td>93.7</td>
<td>99.1</td>
<td>54.4</td>
</tr>
</tbody>
</table>

The results that seem to be the most interesting ones are the ones issued from the stiffness maximizing study and the mass minimizing with a security coefficient of 2.

![Figure 45: Von Mises stresses (left) and elements densities (right) for the stiffness maximisation (up) and the mass minimization (down)](image)

Then, these two studies have been more precisely developed using two parameters of the optimization process named Member Size and allowing the limitation of the elements’ size during the calculation.
First, the Minimum Member Size defines the minimum size of the elements issued from the calculation. It leads to a better simplification of the final geometry. Its value has to be between three and twelve times superior to the average mesh elements’ size. The value here has been defined as 8mm as long as the mesh elements’ size is between 0.8 and 2mm.

![Figure 46: Minimum Member Size (Altair)](image1)

The second criterion is the Maximum Member Size which has the opposite effect limiting the creation of elements with a too important size. It leads to a more lightened geometry but can although provoke fragilities with problems of poor density. This parameter has to be two times superior to the Minimum Member Size and six times superior to the mesh elements’ size: it’s been fixed at 16mm.

![Figure 47: Maximum Member Size (Altair)](image2)

The Minimum Member Size parameter gave an interesting result in the case of the mass optimizing with the creation of a reinforcement between the arms of the triangle, a particularly fragile zone. The average Von Mises stresses maximum is although decreased thanks to this parameter. On the other hand, the Maximum Member Size parameter has been deactivated as long as it was increasing the stresses and not leading to an interesting material removal.

For the stiffness optimization these two parameters leaded to important mass reductions but provoked as well an overflow of the yield strength: then they have not been retained.
<table>
<thead>
<tr>
<th>Study</th>
<th>Minimize the mass</th>
<th>Maximize the stiffness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum Member Size</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Maximum Member Size</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>% Yield strength</td>
<td>66.3</td>
<td>50.3</td>
</tr>
<tr>
<td></td>
<td>134</td>
<td>125</td>
</tr>
</tbody>
</table>

Figure 48: Mass minimization with (left) and without (right) Minimum Member Size

A simple visual analysis allows to see the difference between the two types of optimizations: the result issued from the stiffness constraint shows an important material removal when the one issued from the mass constraint seems to be more robust especially in the area of the triangle's arms submitting important efforts.

The following part of the topological optimization study consists in the redesign of the part with SolidWorks in order to modify the geometry and to better approach the results given by HyperWorks. It's then necessary to verify the Von Mises stresses' state in the part when it's submitted to the predefined static efforts. These stresses need to be inferior to the material's yield strength. This analysis can be realised on HyperWorks, ANSYS or Inspire.

In both cases, many redesigning phases have been necessary to obtain a correct stresses' state by strategically adding material in weakened zones. In fact, the geometry, as it’s proposed by HyperWorks, exceeds the yield strength because of too important material removals and sharp angles. These defects are principally localised around the hub carrier fixation where the part’s lightening is excessive: they have been reduced at the maximum to approach the material's yield strength. There are still some punctual exceedance of this limit in this zone that could lead to plastic deformations of the triangle. Only a test in real conditions could confirm or not the resistance of the triangle.
The topological optimization final results of this suspension’s triangle show a possibility of a total mass reduction on the vehicle between until 2.11kg with the stiffness maximizing calculation and 1.33kg with the mass minimizing one. This result is considerable for a small electric city vehicle as the Twizy: it could allow to raise its autonomy and driving dynamism.

However it has to be noticed that the static simulations realised here can be sufficient to validate the optimised geometry. In fact, as being a part of the suspension system, this triangle will suffer vibrations and efforts provoking its fatigue as flexion cycles. Before making a real endurance test with a prototype it’s interesting to analyse the part modelling its dynamic behaviour. This simulation allows to verify its resistance to impacts, its behaviour during a crash or to know its vibration modes.

For example, an analysis of the five first eigenmodes has been realised with the initial triangle and the one issued from the two different optimizations. They showed a general decrease of the natural frequencies and especially of the fundamental mode. This one is reduced by 20% with the mass optimization and by 40% with the stiffness one. There is definitely a weakening issued from both topological optimization processes that could lead to a premature break of the part.
### Frequencies (Hz) – Eigenmodes

<table>
<thead>
<tr>
<th></th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Original part</strong></td>
<td>76.8</td>
<td>337</td>
<td>520</td>
<td>904</td>
<td>965</td>
</tr>
<tr>
<td><strong>Result from the mass optimization</strong></td>
<td>60.3</td>
<td>277</td>
<td>446</td>
<td>772</td>
<td>940</td>
</tr>
<tr>
<td><strong>Result from the stiffness optimization</strong></td>
<td>44.5</td>
<td>272</td>
<td>359</td>
<td>796</td>
<td>940</td>
</tr>
</tbody>
</table>

Then, it could be interesting to continue this study looking for a balance between a significate mass decrease and a more important respect of the original part’s Eigenmodes. It could be then possible to confirm the part resistance with a complete simulation before the realisation of a validation prototype.

It’s although be seen that this additive manufacturing process was leading to the mechanical properties’ anisotropy. It would be relevant to analyse the effects of some heat treatments on this inhomogeneity. In fact, the following study will show the anisotropy’s evolution with mechanical characteristics and microstructures that could be finalised with mechanical tests.

An ultimate necessary point for the validation of this reconception study is the technical and financial feasibility. It’s important to study the market to know if the dimensions and geometry of the part agree with what is actually possible to produce. It’s although interesting to realize a cost study and to simulate the productions times to know the investment represented by the purchase of machines and the buy of powder for a serial production.
Chapter 4: Heat post treatments study

1 Introduction to the materials

Heat post-treatments’ study has been realised on three different metallic materials distributed by EOS: the 316L stainless steel, the aluminium alloy AlSi10Mg and the nickel superalloy Inconel 718. The additive manufacturing technology used to produce the parts is the Direct Metal Laser Sintering one distributed by EOS too. The laser’s powers used for this materials go from 200 to 400 Watts with a beam diameter from 100 to 500 micrometres.

For each material, a 2.5*50*200mm plate has been ordered. This plates are horizontally produced and haven’t been heat-treated. However a shot peening process has been realised as well as an electrical discharge machining to cut the supports. They will be cut in various samples to realise the experiment design with the different treatments and analysis.

![Ordered plate](image)

Figure 50: Ordered plate

Before and after each step of the heat treatments, various visual and mechanical analysis have been made like the Vickers microhardness at the surface and at the heart as well as the microstructure evolution with optical and electron microscopes. The roughness and topography of the original sample has although been measured before any heat treatment to evaluate the quality of the part’s surface state.
2 Introduction to the heat-treatments

Two types of heat treatments have been made on the three materials: stress relieving ones and a precipitation hardening by solution and age.

Stress relieving treatment:

These treatments are done to eliminate the internal stresses inducted in the parts during the additive manufacturing process. They are mainly due to the temperature gradients or the heat cycles caused by the laser melting. In fact, the laser, solidifying the material layer after layer, leads to a return in fusion of a part of the already melted powder and, thus, an inhomogeneous cooling process. These thermal gradients cause inhomogeneous plastic deformations and the residual stresses: the expansion and contraction thermal cycles of the solidified material can exceed its yield strength.

This treatment is realised to avoid or at least reduce the dimensional change and the risk of fissuring of the parts during the use or manufacturing process. For example, the metal additive manufacturing process can lead to a bending of the parts when they are separated from the supports. The heterogeneity of the material’s thermal history although comes from its capacity to conduct the heat. The non-melted powder in which the part is manufactured acts as an insulator when the supports and the layer conduct the heat. That explains the thermal gradients oriented in the manufacturing direction.

This annealing process won’t modify the material structure and consists in a slow heating followed by a holding time and then a slow cooling process. It should modify the material’s hardness decreasing when the stresses are relieved. The alloy is heated at a temperature allowing the reorganisation by diffusion and relaxation mechanisms without any transformation of the phases. The slow cooling process in necessary to avoid the emergence of new stresses.

Precipitation hardening treatment:

This treatment is done to make improve the material by increasing its mechanical characteristics and its resistance to oxidation and corrosion. It is composed of three steps:
the put in solution, the quenching and the ageing. The ageing step is the progressive hardening of the material until a maximum that can’t be exceeded to avoid a weakening.

This ageing, if done naturally, needs a long process at ambient temperature. The artificial one uses a heating and a holding time, it can be realised directly after the quenching or after a rest of the part at ambient temperature.

The quenching can be realised in hot or cold fluids, the faster it is the better the mechanical strength will be. Its rapidity is defined with the transfer and the cooling time.

The first step improves the ductility of the material making more homogeneous its microstructure: its goal is to erase the thermal history.

A literature review leaded to the following parameters for the three materials used in additive manufacturing or conventional manufacturing processes.

<table>
<thead>
<tr>
<th>Stress relieving</th>
<th>Put in solution</th>
<th>Ageing</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>316L</strong></td>
<td>650°C 2h + slow cooling in furnace</td>
<td>1050°C 1h + water quenching</td>
</tr>
<tr>
<td><strong>AlSi10Mg</strong></td>
<td>300°C 2h + slow cooling in furnace</td>
<td>510°C 6h + water quenching</td>
</tr>
<tr>
<td><strong>Inconel 718</strong></td>
<td>750°C 3h + slow cooling in furnace</td>
<td>950°C 1h30 + water quenching</td>
</tr>
</tbody>
</table>

*Figure 51: Ageing treatment for Inconel 718 (LE COZ)*
3 Samples preparation

For each material, 5 lots have been defined to study the influence of these two heat treatments step by step:

1- Untreated sample
2- Stress relieving treated
3- Put in solution
4- Full precipitation hardening: solution and age
5- Stress relieving treated followed by the precipitation hardening

The samples are prepared as explained in the Appendix 4: cut, enveloped, polished and then etched to reveal the microstructure: grains and weld seams.

![Figure 52: Cut and enveloped samples](image)

The etching are the following ones:

- Stainless steel 316L and Inconel 718: Electrolytic attack at 2.5V during 15 seconds in a solution composed at 10% in mass of oxalic acid diluted in water
- AlSi10Mg: Etching during 10 seconds in Keller’s reagent:
  - 190ml of water
  - 5ml of nitric acid
  - 5ml of hydrochloric acid
  - 2ml of hydrofluoric acid
4 Analysis of the non-treated parts

4.1 Topography and roughness

The roughness has been measured at the surface with the Altisurf measuring station on squared surfaces of $1\text{mm}^2$ and profiles in the length and the width of 100 and 50mm.

<table>
<thead>
<tr>
<th>Material</th>
<th>316L</th>
<th>AlSi10Mg</th>
<th>Inconel 718</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra (μm)</td>
<td>12.9</td>
<td>22.9</td>
<td>9.3</td>
</tr>
<tr>
<td>Sa (μm)</td>
<td>9.8</td>
<td>16</td>
<td>9.4</td>
</tr>
</tbody>
</table>

The aluminium’s roughness is more important than the others’ ant that can be explain by the fact that the samples have been shot peened and that this material is more ductile. It’s less hard and, thus, suffers more the effect of the shot peening than the nickel superalloy for example with a toughness two times less important.

The topography study allowed to precisely measure the non-flatness of the plates that can be observed by naked eye. This bending as explained earlier is due to the internal stresses of the material not eliminated here by a specific heat treatment.

The Radius of curvature $R$ is measured in the length of the plates and is function of the Height and the Width, the smaller is the radius is the more important is the curvature:

$$R = \frac{\left(\frac{W}{2}\right)^2 - H^2}{2H} + H$$

<table>
<thead>
<tr>
<th>Material</th>
<th>H (μm)</th>
<th>W (mm)</th>
<th>R (m)</th>
<th>Thermal conductivity (W/m/K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>20</td>
<td>49.6</td>
<td>15.4</td>
<td>16</td>
</tr>
<tr>
<td>AlSi10Mg</td>
<td>257</td>
<td>49.9</td>
<td>1.21</td>
<td>160</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>6.6</td>
<td>49.4</td>
<td>46.2</td>
<td>11</td>
</tr>
</tbody>
</table>

The aluminium plate is the more curved and by far that can be directly observed. The measure revealed a curvature more than ten times bigger than the other materials. This alloy is less hard than the others and conducts better the heat. This propriety obliges to use a
higher laser’s power in the laser melting process and this leads to an important internal stresses’ state causing an important curvature as long as the aluminium is more ductile.

The topography analysis although allows to get visual results like the curvature in the width of the Inconel plate here.

![Curvature of the Inconel plate](image)

As in the precedent study with the tensile-test pieces, the density has been calculated by measuring the dimensions and the mass of the plates and has been compared to the theoretical one for pieces issued from a conventional manufacturing.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm³)</th>
<th>Density with conventional manufacturing (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>7.5</td>
<td>7.9-8</td>
</tr>
<tr>
<td>AlSi10Mg</td>
<td>2.6</td>
<td>2.7</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>8</td>
<td>8.2</td>
</tr>
</tbody>
</table>

The densities are lower with the additive manufacturing, with an average difference of 4% because of the porosities at the heart of the material and the difficulty to well respect the numerical model. The process is not perfectly precise and this difference has to be considered in order to well reproduce the parts: an oversizing is then necessary.

### 4.2 Microscopy

At first, the different powders have been analysed with the Scanning Electron Microscope for the three materials. Thus, it has been possible to observe and measure the particle size distribution. The stainless steel powder is composed of spheres which diameter is of a few dozens of micrometres on a homogeneous bottom with smaller particles. The same aspect is
observable with the Inconel powder and the aluminium is less homogenous with really smaller spheres and some agglomerations.

Then, the surface of the different non treated samples has been analysed with the SEM what allowed to see the weld seams more or less wide depending on the material.

The stainless steel doesn't allow a precise visualisation of these imprints comparing to the aluminium one for example where they are much more evident with cavities separating them.

The steel and Inconel samples have a similar imprints’ width when the aluminium one is wider. It can be explained with the better thermal conductivity for this material.
The study in the cut realised with the optical microscope after the etching allows to see the manufacturing strategy with the weld seams from the laser’s melting process.

This seams are the thermal affected areas by the laser scan, these molten pools are under the form of circles arcs when seen by profile and ellipses when seen in the plane.

Many measures are possible on this seams and can be related to the manufacturing parameters considering the process as optimized.

For example, the affected depth with the laser scan can be measured in the cut with the depth of the seams and corresponds to the molten pool. It allows to conclude that the laser scans don’t only impact the last layer deposed but although put back in fusion the precedent ones.

In this cut, the layer depth can although be analysed with the distance between two superposed seams.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{Fig_55.png}
\caption{Steel (a), Aluminium (b) and Inconel (c) surfaces: measure of weld seams’ width}
\end{figure}
With both of the views, it’s possible to get information about the average width of the weld seams and to check the coherence with the precedent measure done in surface with the SEM. This value can be related to the laser’s beam diameter.

The surface view gives information about the lasing strategy with the angle between the scans of the laser layer after layer. The one used here is the same as in the case of the tensile-test species, the one using an angle of 67°.

Figure 56: Stainless steel: measures of weld seams’ depth (a), layers’ depth (b and c), weld seams’ width (c and d) and lasing angle (e)
Figure 57: Aluminium: measures of weld seams’ width (a and c), layers’ depth (b) and lasing angle (d)

Figure 58: Inconel: measures weld seams’ width (a, b and c), layers’ depth (a) and lasing angle (d)
The results from the different measures are the following ones:

<table>
<thead>
<tr>
<th></th>
<th>Powder’s diameter (μm)</th>
<th>Weld seams' width (μm)</th>
<th>Weld seams' depth (μm)</th>
<th>Layers’ depth (μm)</th>
<th>Lasing angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>34</td>
<td>93</td>
<td>100</td>
<td>35</td>
<td>69</td>
</tr>
<tr>
<td>AlSi10Mg</td>
<td>30</td>
<td>194</td>
<td>130</td>
<td>28</td>
<td>69</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>35</td>
<td>86</td>
<td>80</td>
<td>42</td>
<td>66</td>
</tr>
</tbody>
</table>

4.3 Hardness of the untreated samples

For each material, Vickers microhardness measures have been realised at the surface and in the cut for every step of the heat treatments. The average values with the standard deviations are done below.

A measure has although been made to get the evolution of the hardness with the distance to the upper surface of the part, the last manufactured layer. This was made to evaluate the effect of the shot peening process. It was not be revealed as long as here the shot peening is used to improve the surface’s state and not the mechanical characteristics to get a better resistance to the fatigue.

The average values before the heat treatments come from measures realised the more precisely possible at the centre of the weld seams. After the stress relieving treatment it was still possible to measure this way but after the put in solution, first step of the precipitation hardening treatment, the seams disappeared. The homogenizing effect was not the same for every material and that caused a decrease of the measures’ repeatability: that’s what will be seen later with the final hardness’ study.

The measuring method is the Vickers type HV0.1 with a load of 0.1kg or 980.7mN during 10 seconds.

A more precise analysis has been realised at heart in the cut before any heat treatment to get information about the variation of the hardness depending on the targeted area in the microstructure. To be able to precisely measure at heart or in the edge of the seams, the load is reduced by ten to 98.07mN or HV0.01.
A literature review for the three materials produced with a conventional manufacturing process made possible the comparison. The parts have not been stress relieved and that can explain the important hardness for these materials, increased by the internal stresses. Although, it will be seen that the targeted areas, the seams hearts, are the harder parts of this inhomogeneous microstructure.

<table>
<thead>
<tr>
<th>Vickers Hardness (HV)</th>
<th>Cut measure</th>
<th>Surface measure</th>
<th>Average value</th>
<th>Conventional manufacturing</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>232.5 ± 22.4</td>
<td>231.4 ± 28</td>
<td>232</td>
<td>120-140</td>
</tr>
<tr>
<td>AlSi10Mg</td>
<td>146.4 ± 9.9</td>
<td>143.8 ± 3.8</td>
<td>145.1</td>
<td>80-95</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>350.1 ± 27.3</td>
<td>325.5 ± 20.8</td>
<td>337.8</td>
<td>210-420</td>
</tr>
</tbody>
</table>

The aluminium sample, more ductile than the others, showed a better homogeneity and made possible a relatively good repeatability of the measures with uniforms imprints and a good visual definition of the different targeted areas.

Thus, the more precise analysis of the microstructure’s hardness has been realised with the aluminium in three different areas of the seams seen by profile:

- 1: the border of the weld seam separating two layers where the grains are bigger
- 2: the Thermal Affected Area, the heart of the weld seams.
- 3: the area close to the upper border that will suffer an annealing with the laser melting process on the next layer.

*Figure 59: Vickers hardness indentations in the different studied areas*
At the borders of the weld seams, bigger grains have been identified, they are due to a slower cooling of the material as they are more distant to the surface than the heart of the seam for example. The Vickers hardness measures here is lower than the average value issued from the precedent analysis. This can be explained with the grains’ size, as long as a grain grows, it resists less to the material’ stresses and its hardness decreases.

In the area 2, the centre of the weld seams, the cooling process is faster and thus the grains are smaller: that explains the higher hardness in this area. This value should although be close to the one measured in the precedent study but is finally higher. That can be explained with the difference of precision of the methods: HV0.1 and HV0.01: in this case it’s more precise to target a specific area but more difficult to measure the diagonals of the imprints then.

The third area can suffer an annealing or even a comeback in fusion when the laser passes to melt the next layer. It has be seen before that the laser doesn’t only thermal affect the last layer but although the already solidified powder. This supplementary energy allows the grains’ growth and give them a hardness’ decrease compared to the one of the second area.

It has been confirmed that a part issued from an Additive Manufacturing process is inhomogeneous with its microstructure composed of weld seams and depending on the exposition of the material to the laser. This specific microstructure leads to the
inhomogeneity of the mechanical characteristics like the hardness. The post heat treatments are made to erase this thermal history and reduce this inhomogeneity.
5 Effects of the heat treatments

5.1 Effect on the microstructures

After the stress relieving treatment, the microstructure should not show big changes except maybe a growth of the grains with the elimination of the internal stresses.

That’s what confirmed the samples with the imprints of the laser melting process still visible but no change of the grains’ size has been visible. These are still submicronic at the centre of the weld seams, this really small size is due to the fast cooling of the laser melted area, the heat is precisely localised while the rest of the part diffuses it.

![Image of microstructures](image_url)

*Figure 60: Optical microscopy at the surface (up) and in the cut (down) after the stresses relieving treatment for the steel (left), aluminium (centre) and Inconel (right)*

The microstructure’s change begins to be visible after the put in solution treatment. The manufacturing process’ marks disappear and are replaced by more conventional microstructures for these materials. However, it’s still possible to observe grains’ anisotropies with their size and orientation as it will be explain.

With the stainless steel sample, the heat at 1050°C called austenitizing, make the grains grow. This austenitic state of the material is frozen with the quenching but in this case a clear anisotropy is still visible. In fact, the grains get the shape of columns that can be seen in the cut, oriented in the direction of manufacturing, aligned on the thermal gradient. These columns are long of about 100 micrometres and wide of about 10. The homogenising effect of the
heat treatment is unperfected here and this anisotropy will be confirmed later with the hardness’ study.

![Figure 61: Optical microscopy at the surface (left) and in the cut (right) after the put in solution of the steel](image)

For the aluminium, after the put in solution treatment, no anisotropy is visible, the microstructure is well homogenised. Spherical precipitates are visible of a few micrometres, these ones are more and smaller close to the surface where a faster cooling process allows better their initiation but not their growth by globularisation.

![Figure 62: Optical microscopy at the surface (left) and in the cut (right) after the put in solution of the aluminium](image)
5.2 Effect on the hardness

This study has been realised on the 5 types of samples in the cut and at the surface:

1. Untreated sample
2. Stress relieving treated
3. Put in solution
4. Full precipitation hardening
5. Stress relieving treated followed by the precipitation hardening

<table>
<thead>
<tr>
<th>Sample</th>
<th>Vickers hardness (HV)</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AISi10Mg</td>
<td></td>
<td>316L</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Surface   Cut</td>
<td>Surface Cut</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>143.8 ± 3.8</td>
<td>146.4 ± 9.9</td>
<td>231.4 ± 28</td>
<td>232.5 ± 22.4</td>
</tr>
<tr>
<td>2</td>
<td>95 ± 11.3</td>
<td>97.3 ± 3.3</td>
<td>215.6 ± 8.1</td>
<td>220 ± 14.3</td>
</tr>
<tr>
<td>3</td>
<td>82.3 ± 4.6</td>
<td>85.9 ± 5.6</td>
<td>200.2 ± 12.2</td>
<td>176 ± 24</td>
</tr>
<tr>
<td>4</td>
<td>99.1 ± 2.2</td>
<td>97.7 ± 5.5</td>
<td>199.3 ± 29.4</td>
<td>182.2 ± 23.9</td>
</tr>
<tr>
<td>5</td>
<td>102.2 ± 6.5</td>
<td>102.2 ± 2.6</td>
<td>194 ± 25.7</td>
<td>179.4 ± 15.9</td>
</tr>
</tbody>
</table>

The aluminium showed a small anisotropy of the hardness between the measures in the cut and at the surface. This anisotropy was not visible with the microstructure: the global small size of the grains make it difficult to compare the two samples as it has be obviously done with the stainless steel. The stress relieving treatment leads to an important drop of the
hardness by eliminating the internal stresses. This effect is not visible with the microstructure’s observation but the mechanical characteristics are changed. Then, the precipitation hardening, as it is called, makes the hardness increase and seems to eliminate the anisotropy observed after the stress relieving treatment. The first step of this hardening, the put in solution, makes the hardness decrease more than the simple stress-relieving treatment. This step, as it has been already observed, let a more homogenous material by fully reorganizing the material and eliminating the laser melting process’ marks. The microstructure is closer to a conventional one for this material and the hardness decreases to approach the one found in the literature review, around 80-95HV.

The same study with the stainless steel showed the same effect for the step of the treatments, the stress-relieving one, with the decrease of the hardness. The put in solution made it decrease more too with the growth of the grains and the apparition of the columns. The anisotropy observed with the preferential orientation of theses grains has although been identified with the hardness’ results. The columns formed have an evident effect on the mechanical characteristics' anisotropy. This one is lower when measured in the cut because the columns are elongated in this direction, which is the manufacturing one, and more narrow when observed at the surface perpendicularly to this direction. However, this material’s study didn’t allow to see the hardening effect that is suppose to make increase the hardness.
It's possible to conclude on the necessity of a stress relieving treatment of parts issued from an additive manufacturing process and on the interest of a precipitation hardening. The put in solution is really interesting when it enables to homogenise the material and eliminate the laser's fusion imprints.

The realised treatments on the aluminium alloy have been validated as long as their results show the improvement of mechanical characteristics and microstructure. This alloy's study could be continued with mechanical validation's test as mentioned earlier.

In comparison, the treatments realised on the stainless steel and the superalloy showed the persistence of the anisotropy and of the weld seams issued from the laser's fusion. The treatments should then be improved and controlled in those cases, it's been noticed for example that the stress-relieving and ageing treatments are maybe defined with to close temperatures.
Conclusion

The present study of the Metal Additive Manufacturing initiated with a general state of art of the 3D printing with the different processes, using metal or not, the principle metallic materials used and the innovative design methods linked to this technology. Then, the following research made focusing on three interesting points, was based on the most actually used process, the most advanced with its precision and the quality of the produced parts: the melted powder bed by laser. The study has been realised on machines or parts distributed by ReaLizer using the selective laser melting process and EOS using the direct metal laser sintering one. The materials on which the different analysis have been realised are the 316L stainless steel, the aluminium alloy AlSi10Mg, the nickel based superalloy Inconel 718 and the chrome cobalt molybdenum alloy.

At first, the manufacturing process has been studied with the objective of identifying the defects and improving the surface state of the parts. Then the topological optimization, enabling the eco design of a part for the additive manufacturing, has been applied to an automobile part after having realised real tests on the 3D printed material to know its mechanical characteristics. Finally, heat post treatments have been applied to additive manufactured parts to study their effect on the mechanical and microstructural characteristics of the materials.

The study of the manufacturing process focused on the laser’s parameters to better control the fusion: the point to point distance and the exposure time, giving the laser’s speed, and the laser’s power. Four types of defects have been identified: the supercooling, the collapse, the cracks and the poor solidification of the material. The macroscopic identification of these defects is obvious for the supercooling and the poor solidification. Then, the microscopic study at the surface and at heart reveals high porosities rates. For the parts with cracks of collapse effect, this microscopic analysis is necessary to validate or not the state of the parts. In fact, it has been noticed that macroscopically the quality of these parts is close to the correct ones. A refining of the parameters is then necessary to obtain a good balance between the different laser’s parameters enabling to improve the surface state of the produced parts.

The first step of the topological optimization study was focused on the break and mechanical properties analyses of the steel additive manufactured parts. Thanks to tensile tests realised on different manufacturing strategies, the anisotropy and ductile variation have been quantified. It has been noticed a more important elasticity for the additive manufactured
specimens comparing with a more conventional manufacturing process. The topological optimization calculation has then been realised, thanks to the data from the tests, on a suspensions’ triangle using the two main objectives: the mass minimization and the stiffness maximization. It’s then been possible to conclude on mass gains from 25 to 40%. A weakening of the structure has although been noticed: it should be controlled with real vibratory fatigue tests.

The last study was based on the heat post-treatments applied on parts issued from additive manufacturing. The treatments realised here were the stress relieving and precipitation hardening ones. The necessity of the stress relieving treatment has been visualised and measured with curved deformation of the non-treated parts. The metal additive manufacturing process although inducts an inhomogeneous and anisotropic microstructure with the orientation and hardness of the weld seams issued from the laser melted powder. The stress relieving treatments enabled to control the internal residual stresses’ state and the part’s hardness. The precipitation hardening, with its first step of put in solution, enables to reduce the anisotropy due to the thermal gradients frozen in the parts. The specific microstructure of the weld seams disappear to let a more conventional grains’ arrangement. The thermal history issued from the repeated and inhomogeneous melting and cooling phases is although erased with this treatment.

Personally, this internship gave me the opportunity to learn a lot about what could be my work as an engineer by managing this research project. I had to set up the complete study of this innovating manufacturing process that is the metal additive manufacturing. It’s been done with the phases of state of art, search for partners, order of samples and definition of the experimental designs and test protocols. It’s been necessary to precisely define the manufacturing, modelling and thermal and mechanical tests parameters as well as the different types of measures and analyses to realise for the samples’ characterisation. I although had the chance to work in, and sometimes discover, various sectors of activity like the production of biomedical solutions with the dental implants, the engineering department with the numerical simulations and the analysis laboratory with the different tests and characterisations. In this way, this project although brought me additional knowledge on measure and analysis techniques as well as on modelling and simulation software.
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Appendices

Appendix 1: AltiSurf

This contactless measuring station uses the light reflexion to determine the samples’ surface state. A light beam from a DEL (white polychromatic light) passes through an optic fibre until a passive probe with a high chromatic aberration's lens. This lens' role is to vertically decompose the light beam in various monochromatic wavelengths on a specific distance that is the measuring range, here 400 micrometres. Depending on the analysed surface’s topography, some wavelengths will be reflected after contacting the surface. These wavelengths of the spectrum are then interpreted as altitudes.

Figure 63: Altisurf (ITA)

The roughness' study has been realized with profiles according to the norms ISO 4287 and ASME B46.1 with Gaussian filters of 10 or 20mm, 1/5th of the study’s wavelength. At the surface, the used norms are the ISO 25178 and EUR 15178N.
Appendix 2: Optical and Digital Microscopy

Various optical instruments have been used for the samples’ microscopic analysis. The main one is the Inversed Optical Microscope zooming from 16 to 1500 times with a polarized light. An images’ analysis system, Leica, is although used to make the measures.

The second instrument used to make shots and measures of the samples is the 3D Video Microscope. This is a digital microscope without any eyepieces, the result is directly observed on a screen and can be zoomed until 7000 times.

![Optical and Digital Microscopes](image)

*Figure 64: Optical and Digital Microscopes (Nikon, Hirox)*
Appendix 3: Scanning Electron Microscope

From the millimetre to the nanometre, this non-destructive measurement mean allows to study the elementary chemical composition and to make shots of the analysed samples. The characterised surface is impacted by a scanning electrons' beam. The material of the sample reacts by emitting information that will be received by specific detectors.

![Image of Scanning Electron Microscope](hitachi.png)

*Figure 65: Scanning Electron Microscope (Hitachi)*

The “Topography” mode allows to highlight the topography of the observed sample thanks to the secondary electrons’ detectors.

The “Composition” mode allows to the highlight the chemical composition’s differences. The more an area is bright, the more it has heavy elements (atomic number Z). This analysed is allowed thanks to the retrodiffused electrons’ detection.

Finally, it’s possible to identify the different elements composing the material thanks to an EDX detector (Energy Dispersive X-ray spectrometry) receiving the photons (X-ray) emitted by the material after the electronic excitement.

![Diagram of Electron-material interactions](gauge.png)

*Figure 66: Electron-material interactions (University of Glasgow)*
Appendix 4: Samples’ preparation: cutting, enveloping, polishing, and etching

The samples’ preparation process starts with the cutting with the microchainsaw with a rotation’s speed of 3000RPM and an advance speed of 0.300mm/sec.

![Microchainsaw (Struers)](image1)

The samples’ enveloping phase is then done by heating with a black conductive resin Polyfast with the following parameters.

![Heated enveloping machine (Struers)](image2)
Then, comes the polishing phase that is manual in a first time. It’s done with Silicon Carbide disks, grade 120. The next step is automatic with a semi-automatic polishing machine following specific parameters to obtain a mirror-polished state.

**Table 1:** Co-Cr-Mo Prepolishing and Polishing Parameters

<table>
<thead>
<tr>
<th>Co-Cr-Mo</th>
<th>Prepolishing</th>
<th>Polishing</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Step</strong></td>
<td>PG</td>
<td>FG2</td>
</tr>
<tr>
<td><strong>Surface</strong></td>
<td>MD-Piano 220</td>
<td>MD-Largo</td>
</tr>
<tr>
<td><strong>Abrasive Type</strong></td>
<td>DiaPro Allegro/Largo 9µm</td>
<td>DiaPro Dac 3µm</td>
</tr>
<tr>
<td><strong>Lubricant’s type</strong></td>
<td>Eau</td>
<td></td>
</tr>
<tr>
<td><strong>Speed (RPM)</strong></td>
<td>300</td>
<td>150</td>
</tr>
<tr>
<td><strong>Force (N)</strong></td>
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<td>30</td>
</tr>
<tr>
<td><strong>Head’s rotation (°)</strong></td>
<td>&gt;&gt;</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td><strong>Time (min)</strong></td>
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<td>4</td>
</tr>
</tbody>
</table>

*Figure 69: Manual Semi-automatic polishing machines (Struers)*
### 316L

<table>
<thead>
<tr>
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<th>Polishing</th>
</tr>
</thead>
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<td>MD-Largo</td>
</tr>
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<td>Abrasive Type</td>
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<td>DiaPro Dac 3µm, OP-S, 0.04µm</td>
</tr>
<tr>
<td>Lubricant's type</td>
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<td></td>
</tr>
<tr>
<td>Speed (RPM)</td>
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<td>150</td>
</tr>
<tr>
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### AISI10Mg

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<th>Polishing</th>
</tr>
</thead>
<tbody>
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<td>MD-Largo</td>
</tr>
<tr>
<td>Abrasive Type</td>
<td>DiaPro Allegro/Largo 9µm</td>
<td>DiaPro Mol R 3µm, OP-S, 0.04µm</td>
</tr>
<tr>
<td>Lubricant's type</td>
<td>Eau</td>
<td></td>
</tr>
<tr>
<td>Speed (RPM)</td>
<td>300</td>
<td>150</td>
</tr>
<tr>
<td>Force (N)</td>
<td>25</td>
<td>30</td>
</tr>
<tr>
<td>Head’s rotation (*)</td>
<td>&gt;&gt;</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td>Time (min)</td>
<td>1</td>
<td>4</td>
</tr>
</tbody>
</table>
### Inconel 718 Prepolishing and Polishing

<table>
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<th>Lubricant’s type</th>
<th>Speed (RPM)</th>
<th>Force (N)</th>
<th>Head’s rotation (*)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MD-Piano 220</td>
<td>DiaPro Allegro/Largo</td>
<td>Eau</td>
<td>300</td>
<td>40</td>
<td>&gt;&gt;</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>MD-Largo</td>
<td>DiaPro Dac 3µm</td>
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<td>150</td>
<td>30</td>
<td>&gt;&gt;</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>MD-Dac</td>
<td>OP-S, 0.04µm</td>
<td></td>
<td>150</td>
<td>30</td>
<td>&gt;&gt;</td>
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<td></td>
<td>MD-Chem</td>
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<td></td>
<td>150</td>
<td>15</td>
<td>&lt;&lt;</td>
<td>2</td>
</tr>
</tbody>
</table>

(*): - « >> » means that the samples’ holder and the table turn in the same direction
-« «<> » means that the samples’ holder and the table turn in different directions

To clean the samples, it is possible to use ethanol, demineralized water, compressed air pistol or an ultrasounds cell.

The chemical attacks are realized putting the samples in the reagent during a certain time and the electrolytic ones with an electrolytic polishing machine connected to the reagent in which is immerged the sample. The machine makes circulate a current in the reagent.

*Figure 70: Electrolytic polishing machine (Struers)*
Appendix 5 : Shot Peening

The shot peening is a process close to the sanding one. It allows to treat surfaces by impacting them with spherical glass projectiles. The impacts lead to the surface’s stripping but it can although be used to clean a surface, to improve the surface’s state are to induce stresses close to the surface and in this way increase its hardness.

Figure 71 : Shot peening effects (Blastservis)

Figure 72 : Shot peening machine (Wikipedia)
Appendix 6: Electrical Discharge Machining

The cutting process Electrical Discharge Machining is although called electroerosion. Electrical discharges are used to remove material. It can only be used on conductive materials no matter of its hardness. It allows a good precision of 5µm but on the other hand the cutting speed is low.

To get the necessary spark for the cutting process the part has to be immersed in a dielectrical bath and a copper or brass electrode is used.

![EDM machining diagram](https://i.imgur.com/EDMmachining.png)

*Figure 73: EDM machining (Wikipedia)*


Appendix 7 : Vickers Hardness

The Vickers Hardness gives an information about the resistance of the material to the penetration of a harder part, here a squared based pyramidal diamond indenter. The Vickers microdurometer applies a constant force on the sample’s surface by the penetrator during a specific time. Nine types of efforts can be tested in a range from 98.07mN to 19.61N, a Vickers hardness from HV0.01 to HV2. The mainly used, HV0.1 allows to realize measures following the norm NF EN ISO 6507-1.

![Vickers microdurometer](image)

*Figure 74: Vickers microdurometer*

The pyramidal imprint let by the penetrator on the sample allows to determinate its hardness. It has a lateral area S measured in mm² with the diagonals of the imprints and then the hardness is defined as P/S where P is a pressure measured in kgf. The Vickers hardness is defined as a number without dimension, HV.

This microdurometer allows to precisely, reliably and rapidly measure the Vickers hardness on the metallic materials studied here. It although allows to see the microstructure of the parts.
## Appendix 8: EOS Materials

| Typical achievable part accuracy [1], [7] | approx. ±20–50 µm  
|                                           | (±0.0008 – 0.002 inch) |
| - small parts                            | approx. ±0.2 %         |
| - large parts                            | approx. 0.3 – 0.4 mm   
|                                           | (0.012 – 0.016 inch)  |
| Min. wall thickness [2], [7]              | 20 µm (0.8 x 10⁻⁴ inch) |
| Layer thickness                          | 2 mm³/s (7.2 cm³/h)   
|                                           | 0.44 in³/h            |

<table>
<thead>
<tr>
<th>Material composition</th>
<th>Element</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>Fe</td>
<td>balance</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cr</td>
<td>17.00</td>
<td>19.00</td>
</tr>
<tr>
<td></td>
<td>Ni</td>
<td>13.00</td>
<td>15.00</td>
</tr>
<tr>
<td></td>
<td>Mo</td>
<td>2.25</td>
<td>3.00</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>0.030</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mn</td>
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<tr>
<td></td>
<td>Cu</td>
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<td>P</td>
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<tr>
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<td>S</td>
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<td>N</td>
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*Figure 75: 316L (EOS)*
<table>
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<th>Description</th>
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<tr>
<td>Smallest wall thickness</td>
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<tr>
<td></td>
<td>approx. 0.012 – 0.016 inch</td>
</tr>
<tr>
<td>Volume rate [3]</td>
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<td></td>
<td>0.9 in³/h</td>
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<td>Al (balance)</td>
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<td></td>
<td>Si (9.0 – 11.0 wt-%)</td>
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<tr>
<td></td>
<td>Fe (≤ 0.55 wt-%)</td>
</tr>
<tr>
<td></td>
<td>Cu (≤ 0.05 wt-%)</td>
</tr>
<tr>
<td></td>
<td>Mn (≤ 0.45 wt-%)</td>
</tr>
<tr>
<td></td>
<td>Mg (0.2 – 0.45 wt-%)</td>
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<td></td>
<td>Ni (≤ 0.05 wt-%)</td>
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<tr>
<td></td>
<td>Zn (≤ 0.10 wt-%)</td>
</tr>
<tr>
<td></td>
<td>Pb (≤ 0.05 wt-%)</td>
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<tr>
<td></td>
<td>Sn (≤ 0.05 wt-%)</td>
</tr>
<tr>
<td></td>
<td>Ti (≤ 0.15 wt-%)</td>
</tr>
<tr>
<td>Typical achievable part accuracy [1]</td>
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</tr>
<tr>
<td></td>
<td>approx. ± 1.6 – 2.4 x 10⁻³ inch</td>
</tr>
<tr>
<td>- small parts</td>
<td></td>
</tr>
<tr>
<td>- large parts</td>
<td>± 0.2 %</td>
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<tr>
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<td>typ. 0.012 – 0.016 inch</td>
</tr>
<tr>
<td>Volume rate [4]</td>
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<tr>
<td></td>
<td>0.44 in³/h</td>
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<tr>
<td>Material composition</td>
<td>Ni (50 – 55 wt-%)</td>
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<tr>
<td></td>
<td>Cr (17.0 – 21.0 wt-%)</td>
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<tr>
<td></td>
<td>Nb (4.75 – 5.5 wt-%)</td>
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<tr>
<td></td>
<td>Mo (2.8 – 3.3 wt-%)</td>
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<td></td>
<td>Ti (0.65 – 1.15 wt-%)</td>
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<td>Al (0.20 – 0.80 wt-%)</td>
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<td>Co (≤ 1.0 wt-%)</td>
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<td></td>
<td>Cu (≤ 0.3 wt-%)</td>
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<td>Si, Mn (each ≤ 0.35 wt-%)</td>
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<td></td>
<td>P, S (each ≤ 0.015 wt-%)</td>
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<td></td>
<td>B (≤ 0.006 wt-%)</td>
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<td></td>
<td>Fe (balance)</td>
</tr>
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</table>

**Figure 76: AlSi10Mg (EOS)**

**Figure 77: Inconel 718 (EOS)**
Appendix 9 : Heat treatments

The heat treatments have been realised with a muffle furnace which temperature increase can be controlled following a ramp until 1100°C. The temperature has been measured and registered during all the treatments thanks to an external probe and an acquisition software to precisely know the temperature’s evolution. It’s even more necessary during the cooling phases in which the temperature is hardly controlled.

*Figure 78: Muffle furnace*
Appendix 10: Project’s cost

The cost of this project is divided into two parts:

- The staff expenses that are the intern’s salary and its travel costs
- The material expenses that are the samples bought for the study of the heat treatments, the tensile test specimens and the realisation of the tractions tests that have not been possible to realise in the partner laboratory

The rest of the studies have been realised with partnerships contracts: in this way the work at the analyses’ laboratory, the simulation calculations and the study of the manufacturing process didn’t imply additional costs.

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<td>316L Stainless Steel sample</td>
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<td>AlSi10Mg sample</td>
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<td>Inconel 718 sample</td>
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The total cost of this research project is then 8882.32€.