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# Comparison of microstructure features and mechanical properties for additive manufactured and wrought nickel alloys 625

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## Abstract

The microstructure characteristics as well as the mechanical properties of an Inconel 625 alloy obtained by three processes: forging, SLM and LMD, are investigated. For the last two processes, known as "additive manufacturing", the influence of printing parameters is considered as well as the role of possible heat treatments. First, microstructural analyses (SEM and EBSD) underline the presence of columnar dendrites with a very heterogeneous grain size for additive manufactured as-built materials. The microstructures appear highly textured, particularly for SLM ones which are also often finer than the ones obtained by LMD. Heat treatments and particularly a 1h-1100°C annealing is proven to improve the printed parts microstructure and to avoid a drastic decrease in terms of ductility, particularly for LMD parts. The LMD process with controlled laser power, coupled with appropriate heat treatment, finally produces materials with both microstructures and tensile mechanical properties close to or better than those of the wrought alloy.

**Keywords:** Nickel Superalloy, Selective Laser Melting, Laser Melting Deposition, Microstructure, Thermal treatments, Hardness, Tensile properties

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## 1. Introduction

Inconel alloy 625 is used extensively in different fields such as aerospace, chemical, petrochemical and marine industries. This material has shown good mechanical performances for components exposed in severe environments, including high temperature strength, high creep and high temperature corrosion resistances [1, 2, 3, 4].

Inconel 625 is a nickel-based superalloy mainly strengthened by the solid-solution hardening effect of elements such as niobium (Nb) and molybdenum (Mo) in a nickel-chromium matrix. After various thermal aging, this alloy was proved to be sensitive to the precipitation of strengthening intermetallic phases such as  $\gamma''$  ( $Ni_3Nb$ ) and  $\delta$  ( $Ni_3Nb$ ), to carbides ( $MC$ ,  $M_6C$ ) and to minor precipitations (Laves particles, Ti-rich particles, etc.) [3, 4, 5]. Extensive characterizations of wrought Inconel 625 have been performed in order to understand the effect of microstructure evolutions through the precipitation of  $\gamma''$  and  $\delta$  phases on the mechanical properties such as hardness and tensile strengths. These investigations reveal that Inconel 625 mechanical properties are greatly improved after annealing of samples for a temperature range between 650-900°C and for length well-above than 1 hour. This improvement is highlighted by the precipitation of hardening phases ( $\gamma''$  and  $\delta$ ) in the nickel-chromium matrix. Over this temperature range, the mechanical properties decrease with the increasing of the annealing temperature [5, 6].

Therefore, the mechanical properties of this material could be subjected to great variations due to the thermal exposure and the effect of manufacturing processes, implying severe thermal gradients and variations, should be considered with care in terms of microstructure features and mechanical properties. Distinct from the conventional machining processes, that use subtractive methods (SM), additive manufacturing (AM) technologies offer the possibility to more easily create components with highly complex shapes at reduced costs. The common AM technologies used to create parts from metallic powders induce laser melting processes such as the Selective Laser Melting (SLM) and the Laser Melting Deposition (LMD). Both of these AM technologies produce 3-D printed parts directly from CAD (Computer-Aided-Design) models, in a layer by layer mode using laser energy. There are however some differences, especially on how the powder is carry onto the manufacturing substrate. On one hand, for the SLM process, a layer of powder is spread on a support, to be selectively melted by a focused

high-energy laser beam, according to a pre-determined pattern in the CAD data. This process is then automatically repeated with a new layer of powder, spread over the previous layers, and again melted, until the printing of the 3-D part is completed. On the other hand, for the LMD process, the high-energy laser beam and the metallic powder combined with a neutral gas- are simultaneously delivered into the melt pool on the substrate by a coaxial nozzle. The nozzle serves to have the powder flows converge at the same point on the focused laser beam. During the LMD manufacturing, the coaxial nozzle system moves along the x-y plane, to build the surface of the pre-defined geometry; and then, the nozzle increment z, is applied to heighten the structure and create the 3-D parts.

Manufacturing processes similar to LMD have been known with different names such as Laser Rapid Manufactured (LRM), Direct Energy Deposition (DED), Laser Engineering Net Shaping (LENS), and other names, depending on the manufacturers [7]. The LMD technologies offer many advantages including surface coatings and repairs of damaged components by using the laser cladding process [7, 8]. Generally, it is difficult to compare LMD and SLM technologies, because they are complementary. Indeed, the SLM process allows the production of very complex components with a higher dimensional accuracy [9], and less porosity than the LMD one [13]. However, the LMD process is faster than the SLM one and can be used to build wider components, in reason of the extended dimensions of the building chamber. Therefore, the process choice depends on the industrial application that is being targeted and also of the material mechanical properties.

The use of the high-energy laser beam results of a very high cooling rate during the deposition/melting process, which affects the microstructure of the resulting parts [8, 9, 10, 11, 12]. Concerning the Inconel 625 obtained by SLM, observations reveal huge differences in terms of microstructure compared to the well-known equilibrium structure of the wrought materials. Recent studies also revealed that the high cooling rate, which occurs mostly via the substrate, leads to a directional grains growth and afterwards the formation of elongated columnar grains[10, 11, 12]. The grains growth direction is mostly perpendicular to the substrate, and a build direction anisotropy is observed.

In the case of LMD parts, a dendritic microstructure is also observed by other authors in nickel-based superalloys [7, 10, 13]. As a consequence, for both LMD and SLM processes, the laser-printed parts exhibit a strong texture anisotropy that has a significant effect on the mechanical properties. However, the reported val-



ues of yield strength (YS) and ultimate tensile strength (UTS), for both additive manufacturing processes, are higher than the ones observed for wrought Inconel 625[12, 13, 14, 15]. Depending of the industrial application and the corresponding mechanical loadings amplitude and directions, the strong texture of these parts could reduce the mechanical resistance in lifetime operation, with the appearance of a sudden brittle fracture of the material. Therefore, it may be necessary to study the effect of heat treatments on the microstructure and mechanical properties of the SLM and LMD parts. Dinda et al. studied for instance the effects of thermal treatment on the dendritic microstructure of Inconel 625 obtained by direct energy deposition (DED) [7]. The dendritic microstructure modification was only observed after a heat treatment above 1000°C, characterized by the recrystallization (equiaxed) of some grains. Microhardness measurements were therefore done to study the effect of these changes on the mechanical properties but tensile tests were not performed. In recent studies, the effect of heat treatment on the microstructure and hardness of SLM Inconel 625 parts were also studied to lead to better isotropic mechanical properties[12, 16]. As example, Li et al. found a homogeneous microstructure and weak texture for samples heat treated at 1150°C [12]. Effects of heat treatments on the tensile mechanical properties have not yet been investigated.

Impact of the post processing heat treatments on the tensile properties of SLM Inconel 625 were investigated in recent papers. These investigations showed a decrease of the mechanical strengths, following to the increase of the ductility, as the recrystallization annealing was done around 1000°C[16, 17]. However, studies about the microstructural evolution under different heat treatments of laser-powder deposited Inconel 625 parts, and its effect on the hardness and tensile properties are quite uncommon. In addition, a direct extended comparison, using exactly the same protocols, of the microstructural and mechanical properties (including tensile tests) of Inconel 625 parts built by these two widely-used additive manufacturing processes have been rarely performed. Therefore, we seek in this present paper to characterize the microstructure features and mechanical properties of components obtained by LMD and SLM processes, and compare them to the conventional wrought Inconel 625. The microstructural evolution under heat treatments will be revealed by Scanning Electron Microscopy (SEM) observations, coupled with crystallographic and grain morphology textures analyses obtained by Electron Backscatter Diffraction (EBSD). The microhardness and tensile properties will be investigated to highlight the effect of annealing treatments and how to reach an equilibrium grain structure in order to obtain properties similar to

114 those of the wrought Inconel 625.

## 115 **2. Material and experimental procedures**

### 116 *2.1. Material and processes*

Ni	Cr	Mo	Fe	Nb	C	Mn	Si	Al	Ti	P	S
Base	22	9	4	3.5	0.025	0.40	0.40	0.40	0.40	0.010	0.010

Table 1: Chemical composition of wrought Inconel 625 (% in weight)

117 As a reference material and a basis for comparison, we use a wrought Inconel  
118 625, supplied by Goodfellow. The as-received wrought material was pre-annealed  
119 at 900°C during several hours in order to stabilize the microstructure. Its chemical  
120 composition is indicated in Table 1.

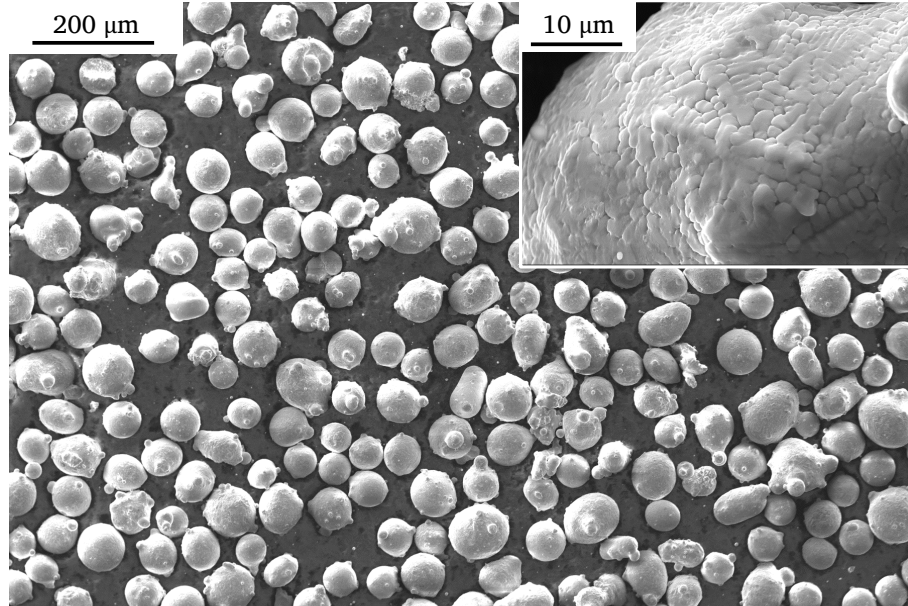


Figure 1: SEM morphology of Inconel 625 powder for LMD, with the upper inset showing micro-dendritic microstructure

121 A commercial Inconel 625 powder was supplied by Oerlikon (MetcoClad625)  
122 for LMD process and its composition is reported in Table 2. Figure 1 shows the

SEM view, illustrating the powder size and morphology. The shape is practically spherical (with some satellites) and the interval particle size is 45-90  $\mu\text{m}$ . The inset in Figure 1 shows the internal microdendritic structure of the powder particle, which characterizes the austenitic  $\gamma$  phase growth.

Ni	Cr	Mo	Nb	Fe	Other
55.0 – 63.0	20.0 – 23.0	8.0 – 10.0	3.0 – 5.0	5.0	2.0

Table 2: Inconel 625 powder nominal chemical composition (% in weight)

LMD parts were fabricated on a BeAM Mobile machine developed by BeAM Company, France, now property of AddUp company. The LMD setup consists of a 500-W fiber laser, associated with a co-axial powder-feeding nozzle and a 5-axis workstation. A defocused laser beam spot was used for powder deposition, and a 30% overlapping ratio between adjacent laser scans was adopted. Argon was used as protective and powder carrier gas. 316L stainless steel 10mm-thick plates are chosen as substrates. The chosen scanning strategy is bidirectional, with a 90° rotation of the laser beam after each deposition as seen on Figure 2.

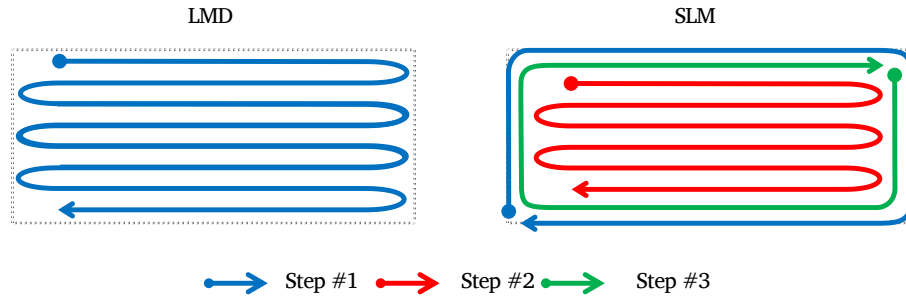


Figure 2: Layer deposition strategy for LMD and SLM built volumes

The output features of the process such as the height and the width of the laser-deposited layer seem strongly dependent on the laser melting deposition (LMD) parameters [18]. The more influent processing parameters on the laser clad characteristics are the laser power  $P$  (W), the scan speed  $V$  (mm/min), and the powder feed rate  $Q$  (g/min). The parameters used in this study to build LMD samples are reported in Table 3

Main parameters	Values	Fixed parameters	Values
P (W)	270, 300, 330	Laser spot (mm)	0.7
V (mm/min)	2000, 2500	Overlapping (%)	30
Q (g/min)	7	Z increment (mm)	0.2

Table 3: Process parameters values used to build LMD parts.

Another commercial Inconel 625 powder was supplied by LPW Technology (LPW625) for SLM process and its composition is identical as the one reported in Table 2. Powder particles are also spherical and their size ranges between 10 and 45  $\mu\text{m}$ . The SLM parts have been produced using the Realizer SLM125 machine installed at ENGIE Laborelec's premises. The optimized processing parameters were adopted for the built Inconel 625 parts, with a laser power of 247W, a scan speed of 1000mm/s, a hatching distance of 0.08mm and a layer thickness of 0.2mm. Once again the scanning strategy is illustrated on Figure 2. The building platform was kept at 100°C to reduce the residual stresses between the substrate and the sample. Inconel 625 bars ( $12 \times 12 \times 70\text{mm}^3$ ) were fabricated by both SLM and LMD processes. The rectangular bars were horizontally printed, parallel to the XY plane as seen on Figure 3.a.

## 2.2. Experimental protocols

Cubic samples were sectioned from rectangular parts and prepared to study the microstructural features. They were polished with SiC paper up to grade 4000, then with diamond paste down to 1  $\mu\text{m}$ . The polished specimens were then electro-etched for 1-3 minutes under 5 V potential, in a solution of 70 mL phosphoric acid and 30 mL water, to highlight the microstructure.

Microstructures were then observed thanks to a digital microscope (Keyence VHX 1000) and a Field Emission Gun - Environmental Scanning Electron Microscope (FEG-ESEM - FEI Quanta 600). EBSD maps with an area of  $1.5 \times 1.5\text{mm}^2$  and a pitch of 1.5  $\mu\text{m}$  were obtained under 25 keV and a spot size of 4.5 (equivalent to a 0.47 nA probe current) with a Nordlys II camera. The post processing of these maps was done with CANNEL 5 software (Oxford Instruments) in order to follow the crystallographic and grain morphology textures evolutions (grain size, orientation) after heat treatments.

To study the effect of the thermal post-treatment on the microstructure, the

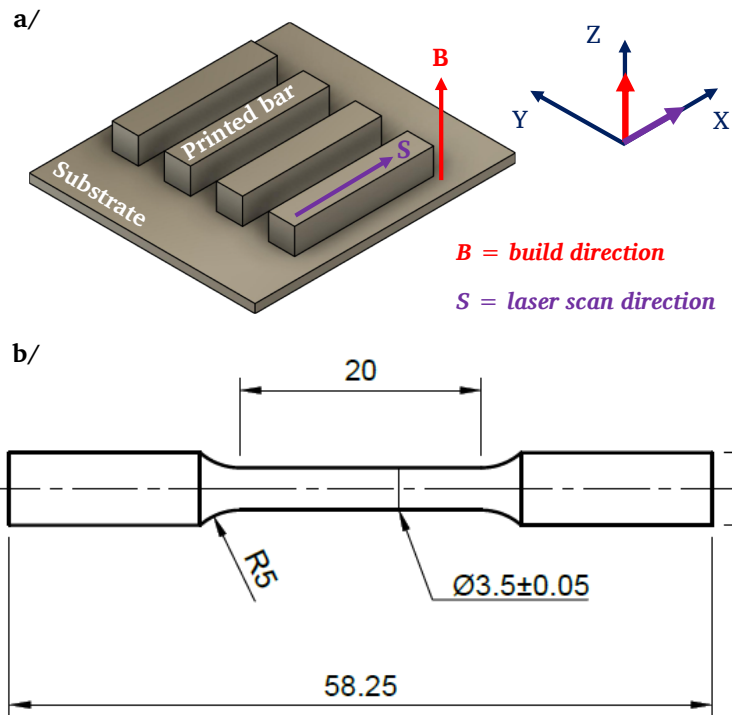


Figure 3: a. Illustration of the rectangular bars, printed by the SLM and LMD processes; b. Geometry of the tensile test specimens, obtained from printed rectangular bars ( $70 \times 12 \times 12 \text{ mm}^3$ )

as-built samples were annealed respectively at 600, 700, 900, 1100°C for 1 hour followed by cooling in air. The lattice parameters, useful to indirectly identify the phase transformations, were measured using a X-ray diffractometer by Cu K $\alpha$  radiation at 40 kV and 100 mA. The standard scanning  $\theta - 2\theta$  geometry was done for all samples from 20°C to 120°C with a 0.01°C step size and 1s dwell time.

The hardness was measured using a Vickers microhardness tester (Mitutoyo, MVK-H1), applying a 0.5kg load for a dwell time of 15s. The final hardness value was obtained by averaging data from twelve indentations on each sample. Tensile tests were finally performed at room temperature, under a constant strain rate of  $10^{-3} s^{-1}$  using an Instron 5960 dual column tabletop testing system and a 30kN load cell. Tensile specimens were machined from horizontal rectangular bars ( $12 \times 12 \times 70 mm^3$ ) according to the geometry specified on Figure 3.b for both LMD and SLM processes, following ISO 6892-1 standard recommendations. Specimens orientation was chosen to characterize tensile properties perpendicular to the build direction and some specimens were heat-treated before tensile tests at 900°C and 1100°C during one hour into a primary vacuum furnace ( $10^{-2}$  mbar), to study the thermal post-treatment effects on the mechanical properties. At least three specimens were tested for each condition to assure statistical representativeness.

### 3. Results and analyses

#### 3.1. Microstructure characterization

##### 3.1.1. Inconel 625 obtained by SLM process

Figure 4 show the transverse section images of Inconel 625 obtained from the SLM process. The microstructure of SLM parts frequently exhibits traces of melt pools, created during the laser beam scanning. The shape and the width of these melt pools depend on the adopted laser scan strategy. The nearly "V" shape is attributed to the Gaussian distribution of the laser energy, with the highest value focused on the center of the laser beam [10]. The microstructure consists on columnar dendrites as a result of the cooling that mostly occurs via the substrate and the deposit. The heat flux direction during the solidification of the melt pool thus enforces the vertical growth of the grains which appear nearly perpendicular to the substrate.

This very fine microstructure can be observed by SEM, as shown in Figure 5. Two kinds of dendritic shape structures can here be observed: cellular-shaped structures, and elongated dendrites, generally called "primary dendrites". These

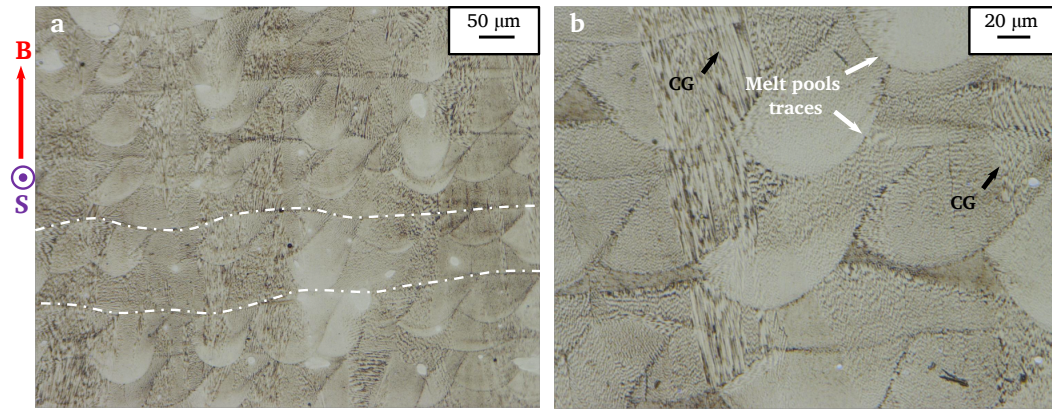


Figure 4: a. Optical micrograph of Inconel 625 processed by SLM at different magnifications (dotted lines are added to emphasize the layers).; b. Optical micrograph of Inconel 625 processed by SLM at different magnifications (CG = columnar dendrites) - B stands for Building direction and S for Laser scan direction

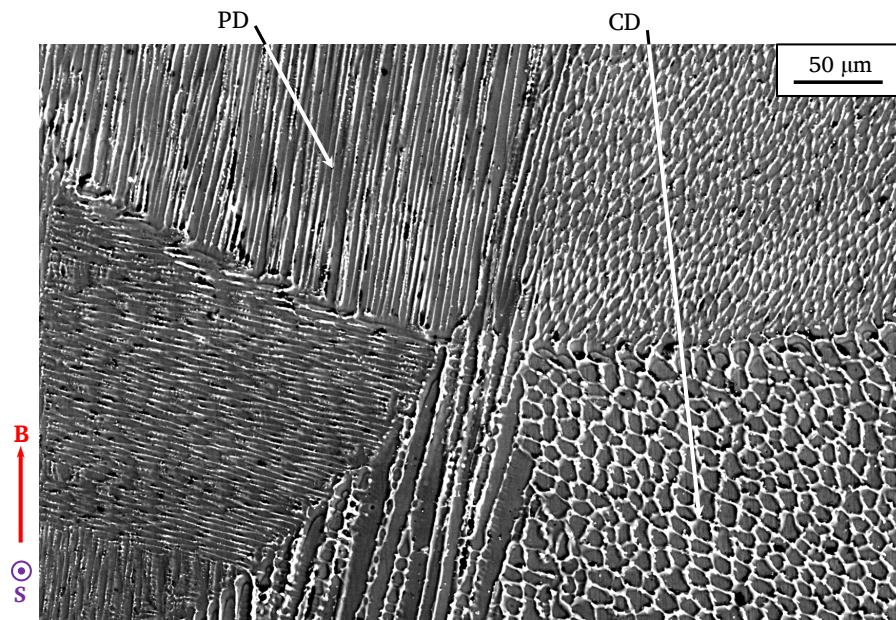


Figure 5: SEM images of Inconel 625 processed by SLM (PD = primary dendrites; CD = cell-like dendrites)- B stands for Building direction and S for Laser scan direction



latter grow in different orientations, depending on the main heat flux direction. Furthermore, the same dendritic structures are observed in the literature on the same kind of material by others researchers [10, 11, 16].

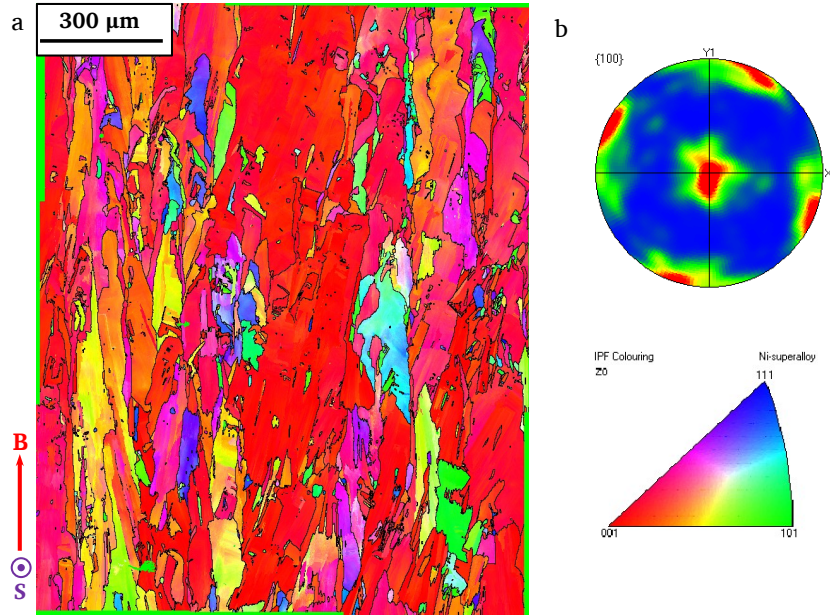


Figure 6: Microstructure of the SLM formed Inconel 625 from EBSD analyses: a. inverse pole figure (IPF-Z) colored map of the Y-Z section; b. {100} pole figure of the map - B stands for Building direction and S for Laser scan direction

EBSD analyses help to reveal the grains crystallography and morphology structure of the as-built materials on Figure 6. The pole figure corresponding to the Y-Z section map exhibits a very strong texture for the SLM sample, quite similar to standard observations for single crystals. This result can also be predicted with the very dominating red color of the IPF-Z colored map. Indeed, grains are aligned along the Z-direction, in consequence of the vertical high cooling flux, which occurs through the building platform. Therefore, the grains grow mostly from the substrate towards the melt pool boundary in a columnar structure. The preferential orientation of the grains is found to  $\langle 001 \rangle$ . This typical texture for the Inconel 625 obtained by the SLM process has previously been observed for similar material [11].



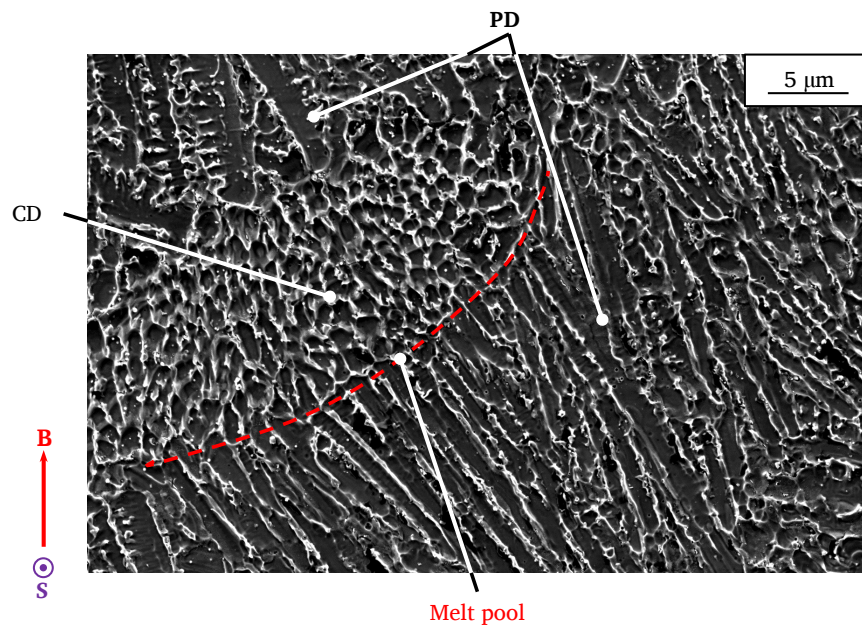


Figure 7: SEM images of Inconel 625 processed by LMD; PD = primary dendrites; CD = cell-form dendrites - B stands for Building direction and S for Laser scan direction

### 3.1.2. Inconel 625 obtained by LMD process

Figure 7 shows the Y-Z section images of the Inconel 625 sample fabricated by the LMD process. The microstructure appears to be also composed of cellular and primary dendrites. The cellular dendrites are often observed in the bottom part of each layer, near the melt pool limit.

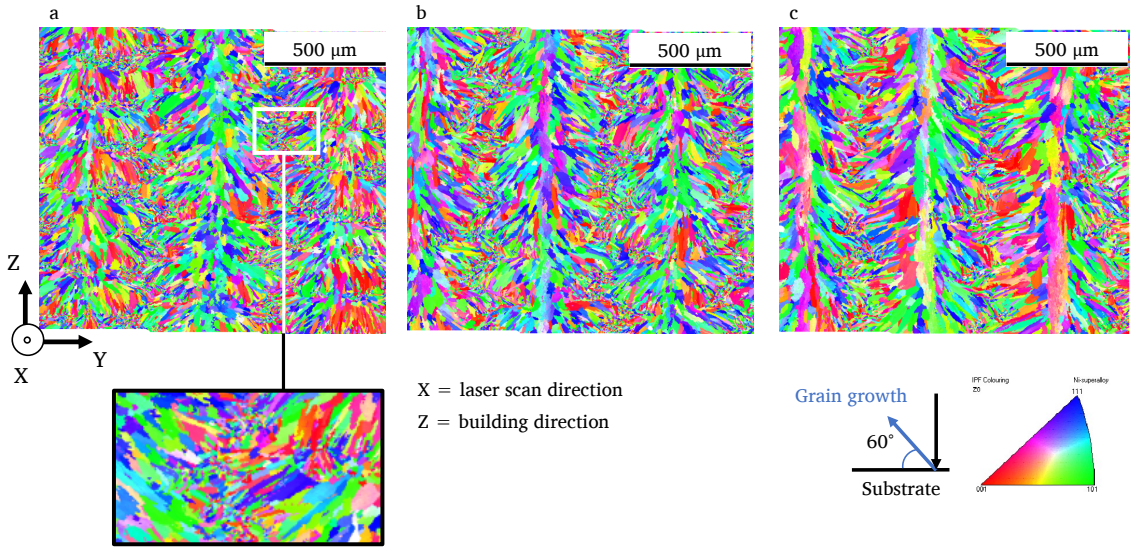


Figure 8: EBSD analyses of the LMD Inconel 625. Inverse pole figure (IPF) of the transverse section observations of samples printed at the same scan speed of  $2500 \text{ mm.min}^{-1}$  and different laser power. (a) 270 W; (b) 300 W; (c) 330 W.

Figure 8 presents the EBSD analyses of the Inconel 625 LMD samples. The IPF-Z images of the transverse section show a random texture of grains, contrary to the SLM samples. Strong differences in produced textures highlight an important relationship between the process and the microstructure. Indeed, the way the manufactured part is cooled down in the different processes is of primary importance to understand the resulting microstructure. SLM parts are in direct contact with the supporting plane but also with the powder bed, reducing significantly the cooling rate as this latter acts as thermal insulator. In the contrary, for LMD specimens, there is an obvious vertical cooling toward the supporting plane but also heat exchanges with the ambient air and most especially an extra cooling coming from the last solidified layers beyond the melting bath. Dinda et al. [7] also states this hypothesis of shared cooling between the substrate and the adjacent

solidified layers in the case of a powder deposition manufacturing process, which would cause grain growth at an angle of less than 90 compared to the substrate and therefore a less pronounced texture. This weak texturing for LMD specimens could therefore be related to a bidirectional growth of the dendrites grains. It has been pointed out in the literature[7, 11], that the  $\langle 001 \rangle$  preferential orientation of grains is a consequence of the vertical heat flux direction, which imposes to the primary dendrites a growth with an angle of about 90° with the substrate, which is the case for SLM specimens . However, primary dendrites of each deposited layer are not perpendicular to the substrate for LMD specimens. In our case, the grain growth in each deposited layer converges to the center area, to form an inverted "V" grain structure as shown in Figure 8. The angle of about 60 (also coherent with values found by Dinda et al.), underlines that the vertical cooling to the substrate could be twice as intense as the horizontal cooling flow via the previously solidified zones.

Figure 8 also show the effect of the LMD manufacturing parameters on the Inconel 625 microstructure. Here, all the manufacturing parameters indicated in Table 3 were kept constant, except the laser power; three value are therefore investigated : 270W, 300W and 330W while the scan speed was taken constant at  $2500 \text{ mm.min}^{-1}$ . Results show that the laser power affects the size and the morphology of the primary dendrites. For the minimal considered power value, 270 W, the primary dendrites appear to be finer than at 330 W. Furthermore, the lower the laser power, the higher the ratio of small grains, located between deposit lines, resulting of back and forth for the nozzle, appear.

These small grains are probably resulting from poorly melted powder or could also be cellular dendrites, observed previously in Figure 7. At 330 W, the ratio of small grains decreases greatly and the size of the primary dendrites increases. These results might be explained by the rise of the melting temperature for the powder with the laser power that could contribute to avoid poorly melted powder. In the same way, a high temperature increases the local thermal heat flux, which leads to much slower cooling rates and thus to significant dendritic growth of grains for high laser powers.

Table 4 shows a statistical analysis of LMD samples microstructures obtained from EBSD maps. For the grains detection, the indicated critical misorientation is 15° and the grain filter (the minimum pixel area) for one grain is chosen at 3 pixels. Here, size of grains is first investigated and a threshold of  $10 \mu\text{m}$  is cho-

Laser scan speed (mm/min)	Laser power (W)	Fraction surface of small grains* (for grain size < 10 $\mu$ m)	Ratio of dendritic structure (elongated grains : AR > 5)*
2500	270	12.2%	4.14%
	300	8.4%	4.72%
	330	3.6%	6.50%

Table 4: Effect of the manufacturing parameter (the laser power), on the grain size and the dendritic microstructure (\*) : estimated on a  $1.5 \times 1.5 \text{ mm}^2$  area, from EBSD analyses.

sen to separate small and big grains (knowing that the average grain size for our reference wrought sample is 15  $\mu$ m). In these conditions, results clearly denote a decrease of the small grains fraction with the laser power. The ratio of dendritic grains is also calculated, by considering the number of grains with an aspect ratio greater than 5. The aspect ratio (AR) is a parameter which allows characterizing the morphology of the grains from EBSD analyses. It corresponds to the ratio between the large and the short length of the ellipse that fits best with the grain. When the grain presents a perfect equiaxed form, AR is about 1, but this perfect value stays unreachable in standard material. Therefore, grains AR distribution is measured for our reference, the previously introduced wrought Inconel 625, to know the repartition of equiaxed grains.

From results shown in Figure 9 and Figure 13.d, the AR critical value was chosen equal at 5. Despite microstructure for wrought material is qualified of equiaxial, the EBSD data of the as-received Inconel 625 reveal some annealing twins, which present an elongated geometry with AR values more than 5. This can explain the non-zero values of the AR distribution beyond 5. Thus, we assume that for AR > 5, the grain shape could be considered as elongated for the LMD samples. Then, the ratio of elongated grains is estimated in percent from EBSD data to investigate the effect of the laser power. Results are presented in Table 4. One can see that the increase of the laser power promotes the formation of elongated grains in the material. The laser power at 330W quicken the dendritic growth of grains whose appear bigger than those at 270W and 300W as observed on Figure 8. The microstructure seems once again narrowly linked to the additive manufacturing process. Therefore, it seems necessary to consider the effect of others manufacturing parameters such as the laser speed, the powder flow rate, the scanning strategy, the nozzle increment in height, the powder qual-

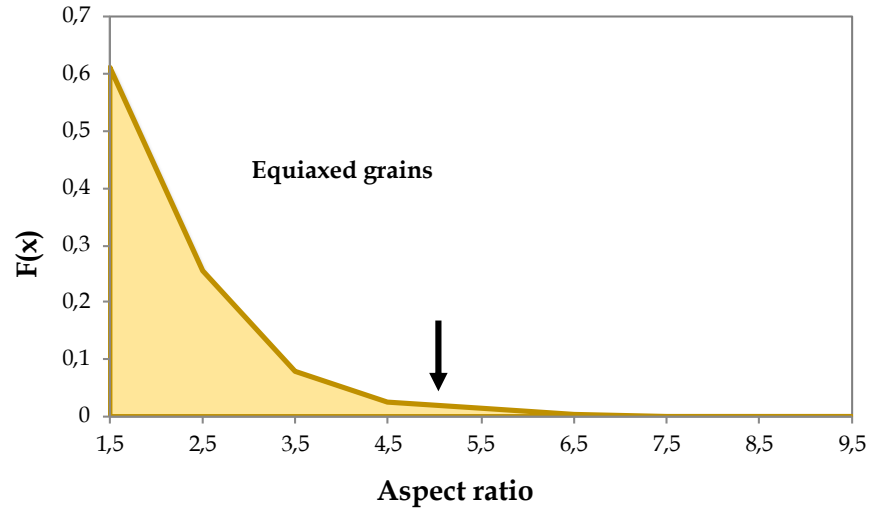


Figure 9: Aspect ratio distribution of grains in the wrought Inconel 625, showing the equiaxed grains repartition. The arrow indicates the critical value of the aspect ratio for an equiaxed structure.

ity and the substrate quality on the metallurgical rendering of the printed material. An optimization of these parameters should enable to reach a microstructure with a quality the nearest as possible as wrought material. Work is underway to then define more suitable parameters.

### 3.2. Effect of heat treatment on the microstructures

#### 3.2.1. Our reference: wrought Inconel 625

First, the effect of heat treatment on the microstructure of the conventional Inconel 625 was investigated to study the phase transformations, independently of the additive manufacturing process. Our reference wrought Inconel 625 was heated at 600°C, 900°C and 1100°C, in a furnace during 1 hour, and then cooled in air.

It has been widely reported in the literature that Inconel 625 consists in an austenitic  $\gamma$ -Ni face-centered cubic solid solution, in which the intermetallic phases such as  $\gamma''$  and  $\delta$  are distributed in the grains and along the grain boundaries, respectively[5, 7]. Depending on the annealing temperature, the intermetallic phases  $\gamma''$  and  $\delta$  may precipitate in the matrix and affect the atoms rearrange-

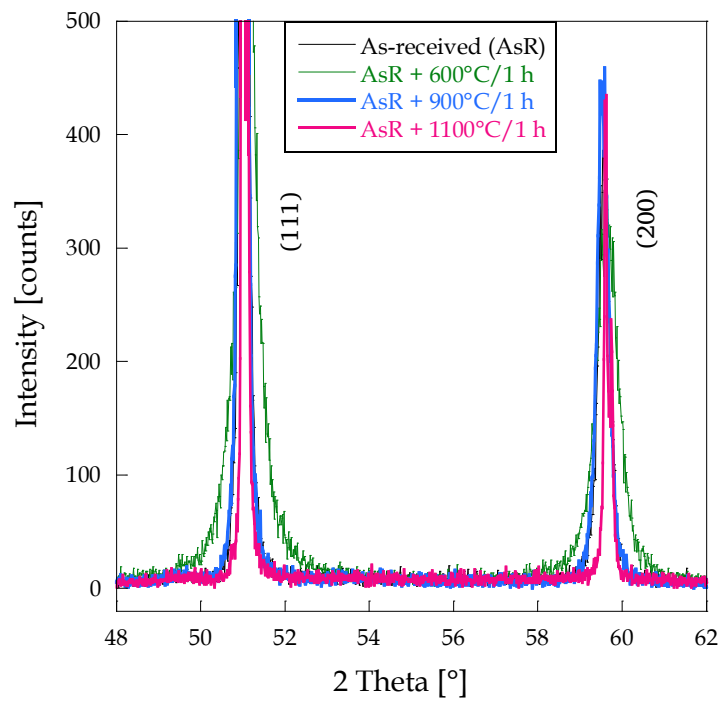


Figure 10: The (111) and (200) X-ray diffraction patterns of the wrought Inconel 625 sample annealed at different temperatures.

ment in the crystal lattice. X-Ray Diffraction (XRD) on the wrought samples under different heat treatments is commonly used[7, 11] to underline the appearance of a new phase in the austenitic matrix as the microstructure characterization is often difficult using standard techniques. Even if intermetallic phases are hardly detectable in the XRD diffraction patterns in reason of their small size[7], the variation of the  $\gamma$ -Ni matrix lattice parameter can here indirectly imply the nature of precipitation. This lattice constant is calculated from the (200) diffraction peak as seen on Figure 10, according to the Bragg diffraction relation.

Treatment of wrought Inconel 625	Lattice parameter(pm)
As-received	360.146
As-received + 600°C / 1h	359.546
As-received + 900°C / 1h	360.145
As-received + 1100°C / 1h	360.000

Table 5: Variation of the lattice parameter of the wrought Inconel 625 under different heat treatments.

Results are shown in Table 5 and are in accordance with previous studies [7, 11]. Without heat treatment, the strengthening elements such as Mo, Nb remain in the  $\gamma$ -Ni matrix and cause a large lattice distortion. When the samples are heated at 600°C, the Nb atoms comes out of the matrix to form the  $\gamma''$  precipitates and the lattice parameter decreases correspondingly. As the temperature increases, the strengthening elements of precipitates are progressively dissolved into the matrix, leading to the increase of the lattice parameter. At 1100°C, while an increase in the lattice parameter was expected, a decrease is obtained. This observation can be explained by the presence of carbides precipitating from the matrix above 1000°C, as shown in literature[11, 12].

Figure 10 shows the diffraction peak positions of the (111) and (200) crystal plane for different heat treatments. A broadening of these peaks can be observed after annealing at 600°C. The higher the annealing temperature, the narrower the integral line-breadth of the peaks becomes. Broadening of the XRD peaks is mainly attributed either to the crystallite size or to the increase of the micro-stresses in the material. In our specific case, we assume that the observed variation comes from the micro-stresses. Micro-stresses are properties of the sample that result from the imperfections in the crystal lattice. These imperfections are

often associated with the local strains occurring on distances less than the crystal dimensions. The precipitation of intermetallic phases in the austenite phase could probably affect the atoms rearrangement in the lattice, leading to a small deformation into the crystal lattice. The micro-stresses variation from one point to another within the crystal lattice may alter the lattice spacing that causes the broadening of the diffraction peak, as seen after annealing at 600°C. These assumptions are corroborated by microscopic observations such as those shown in the Figure 11. At 700°C,  $\delta$  hardening precipitates in globular and acicular form are observed. These precipitates gradually decrease at 900°C until they disappear at 1100°C. These results were compared with those of Radavich et al.[5] with a good consistency.

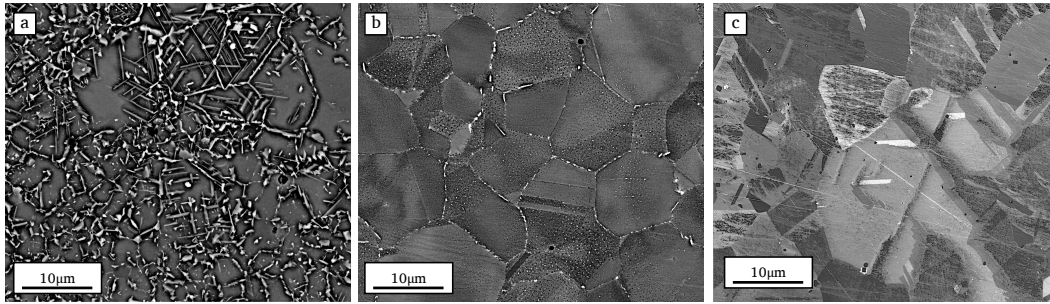


Figure 11: SEM images of wrought Inconel 625 samples annealed 1h a/ at 700°C; b/ at 900°C; c/ at 1100°C;

### 3.2.2. Inconel 625 obtained by SLM and LMD processes

Thereafter, thermal post-treatments were performed at 700°C, 900°C and 1100°C to study the evolution of the dendritic structure observed previously for the SLM and LMD samples. Figure 12 shows SEM images of samples heat-treated at these temperatures. Observations reveal a constancy of the dendritic microstructure after annealing at 700°C for both processes (here only SLM results are presented). The dendritic structure stays stable up to 900°C. At this point, the first steps of the grains recrystallization take place with a widening of the dendrites grains as shown in Figure 12.b for a SLM sample. However, these grains remain elongated in the vertical plane and parallel to the build direction. When the SLM samples were annealed at 1100°C, the equiaxed grains start to appear with annealing twins. These twinning highlight the release of the stored residual stress in the as-built SLM sample, due to the high cooling rates during the building process[12].



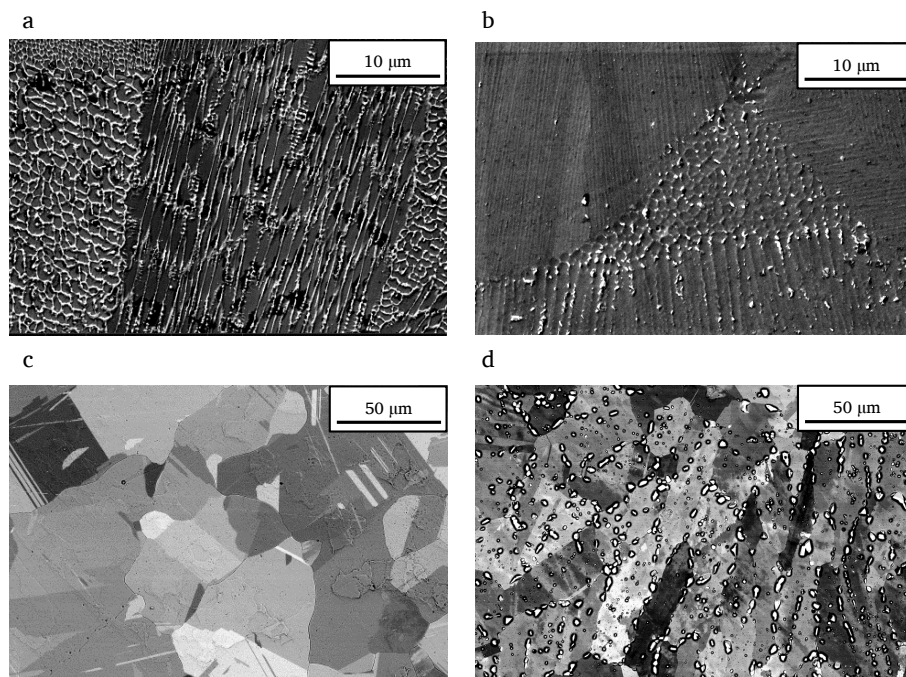


Figure 12: SEM images of the printed Inconel 625 samples annealed 1h at different temperatures. a/ SLM at 700°C; b/ SLM at 900°C; c/ SLM at 1100°C; d/ LMD at 1100°C

Figure 12.c reveals that the recrystallization process is achieved and there are no dendritic grains after annealing at 1100°C for SLM samples, with a microstructure which is close to the one obtained for wrought samples after 1h at 1100°C as seen on Figure 11. Nevertheless, Figure 12.d shows that, in the case of a LMD sample heat-treated during 1 hour at 1100°C, the microstructure consists in a mixture of elongated and equiaxed grains, as an evidence that the recrystallization process is here not completed. Dendrites inclination (about 60° with the substrate as seen on Figure 8) observed in the as-built LMD sample and not in the SLM as-built samples, may explain these results. Further investigations should be here considered. As stated for the wrought material, precipitates can also be observed during the recrystallization process. The chemical nature of these precipitates was not studied in detail. Chlebus et al. performed EDS analyses in these interdendritic spaces for an Inconel 718 and observed "small particles of eutectic mixture ( $\delta$ +Laves-type phase) and carbides" [19]. These results were also found in Antonsson et al.[20]. Indeed, the high melting temperature during the process contributes to raising concentrations of Nb, Mo, and C high enough to promote a rapid eutectic reaction in interdendritic spaces[19], explaining the presence and evolution of these precipitates with thermal history.

The EBSD maps in Figure 13 show the grains morphology of the as-built and heat-treated LMD samples. It underlines that the elongated grains morphology, specific to the dendritic microstructure, is found on the samples as-built and heat-treated at 900°C (Figure 13.a and .b). In both cases, one can observed the inverted "V" shape of the dendrites. As seen in Figure 13.c, the heat treatment at 1100°C begins to eliminate the fine dendritic and columnar grains by recrystallization. However, this annealing is not sufficient to obtain a homogeneous microstructure compared to the as received wrought sample presented as a reference on Figure 13.d. Thus, the recrystallization process seems to be not complete after a heat treatment of only one hour at 1100°C. For the wrought material, the grain size distribution varies from 5 to 40 $\mu$ m, while for the LMD sample heat-treated at 1100°C/1 h, the range is from 5 to 180 $\mu$ m.

### 3.3. Microhardness measurements

Figure 14 shows the microhardness variation of the wrought, SLM and LMD samples, as-built and heat-treated at 700, 900 and 1100°C for one hour. For the as-built SLM and LMD specimens, the hardness values are  $313 \pm 9$  HV and  $315 \pm 14$  HV, respectively. These results are higher than those obtained for the wrought sample, measured at  $260 \pm 5$  HV. The higher hardness of printed samples may be

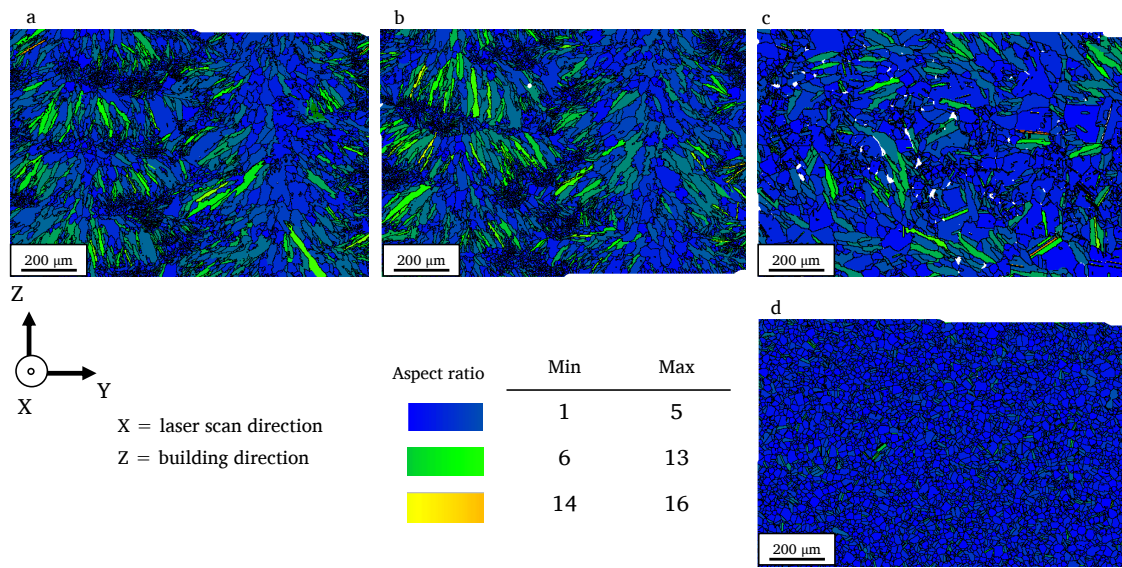


Figure 13: Grains morphology of LMD samples printed for a laser power of 270 W. (a) as built sample; (b) heat treated at 900C; (c) heat treated at 1100C; (d) for comparison, grains morphology of the wrought Inconel 625.

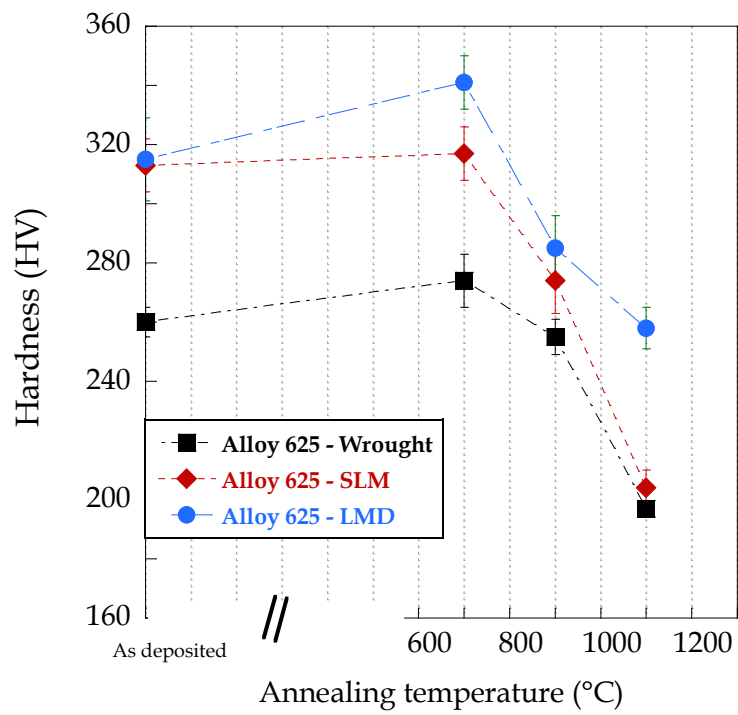


Figure 14: Hardness variation of Inconel 625 samples after annealing at different temperatures

explained by a potential high value for dislocation densities in the material resulting from the high cooling rate of the deposited layer during the printing process ( $10^5 K.s^{-1}$ ). Thus, the important solidification rate contributes to trap the heaviest atoms, i.e. Nb and Mo, within the interdendritic areas, which may contribute to increase the residual stresses in the as-built samples[12].

After post-annealing at  $700^\circ C$ , the hardness increases for all the samples: it can be seen as a marker of the precipitation of the intermetallic phases  $\gamma''$  in the matrix. XRD results presented above are furthermore consistent with this conclusion. Indeed, the hardening precipitates have been indirectly highlighted in the  $\gamma$ -Ni matrix by the lattice parameter calculation in the case of the wrought sample heat-treated at  $600^\circ C$ . As well, the decrease of the lattice parameter observed for the sample annealed at this temperature could be explained by the precipitation of  $\gamma''[Ni_3Nb]$  phases resulting from the exit of Niobium atoms from the crystal lattice [7].

For the SLM and LMD samples, heat treatment at  $700^\circ C$  during 1h leads to a hardness increase of respectively about 1% and 8%. Li et al [12] compared an Inconel 625 processed by SLM before and after an heat treatment at  $700^\circ C$  but during 24h. They show that the release of segregation elements (Nb and Mo) entrapped in the interdendritic regions firstly leads to the decrease of the dislocation density, but even more, causes the precipitation of more  $\gamma''$  phases, increasing slightly the hardness [12]. It seems here that the LMD sample heat-treated at  $700^\circ C$  for 1h also promotes the formation of more strengthening precipitates as  $\gamma''$  phases. The dendritic structure found on the LMD samples seems then to facilitate the departure of Nb and Mo elements, useful to form the strengthening  $\gamma''$  phases, and thus increasing the measured hardness.

For the samples heat-treated at  $900^\circ C$  and  $1100^\circ C$ , results show a decrease of the hardness, certainly due to the progressive dissolution of the  $\gamma''$  precipitates in the matrix. In addition, at  $1100^\circ C$ , grain coarsening highly contributes to this hardness lowering. However, the hardness of the LMD sample heat-treated at  $1100^\circ C$ , remains higher than that of the wrought sample annealed at the same temperature (see Figure 14). On the contrary, the hardness measurements are almost similar for the SLM and wrought annealed specimens. In fact, it can be seen on Figure 12 (d) that some precipitates were still present along the grain boundaries of the LMD sample, despite the high temperature reached during the heat treatments.  $MC$  and/or  $M_{23}C_6$  carbides can then be seen as good candidate to

precipitate along the grain boundaries [12]. On the other hand, these precipitates are not found for the SLM sample post-annealed at 1100°C as seen on Figure 12 (c). The strengthening effect of the LMD sample, heat-treated at 1100°C could therefore be related to these carbides precipitates.

### 3.4. Tensile properties of Inconel 625

Tensile tests were first carried out on the as-received and the annealed-wrought material. Two specimens were used to characterize each annealing condition and the mean values of tensile properties are shown in Figure 15.

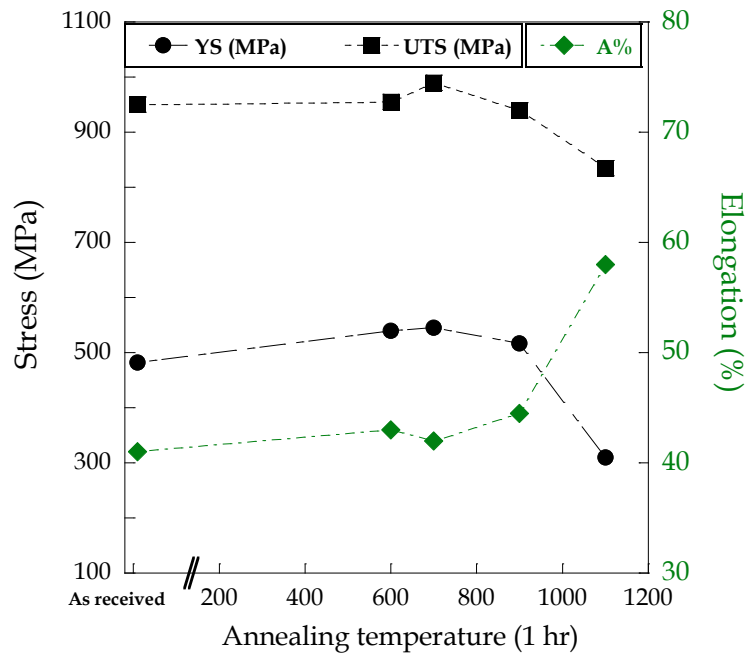


Figure 15: Tensile properties of the wrought Inconel 625 at different annealing temperatures

A small increase of the yield stress (YS) and the ultimate tensile strength (UTS) is observed for specimens heat-treated at 700°C but this best-observed strength characteristics (YS and UTS) are followed by a slight decrease of the elongation to failure at this temperature, revealing that heat treatment at this temperature range does not greatly affect the ductility of the material. These pretty

461 good tensile properties are probably due to the precipitation of the strengthening  $\gamma''$  phases after exposure at temperatures between 600 and 700°C. Tensile  
 462 strength characteristics (YS and UTS) then decrease as the heat treatment temper-  
 463 ature increases. For specimens heat-treated at 900°C, this decrease is the result  
 464 of a progressive dissolution of the precipitates in the matrix. Simultaneously, the  
 465 ductility of the Inconel 625 heat-treated at 900°C and 1100°C increases gradually.  
 466 The heat treatment was not optimized here because it was just a question of spec-  
 467 ifying the influence of exposure to high temperatures on the material behavior in  
 468 simple traction. Subsequently, a compromise can be sought in terms of heat treat-  
 469 ment to obtain high tensile properties combined with good elongation to failure.  
 470

471

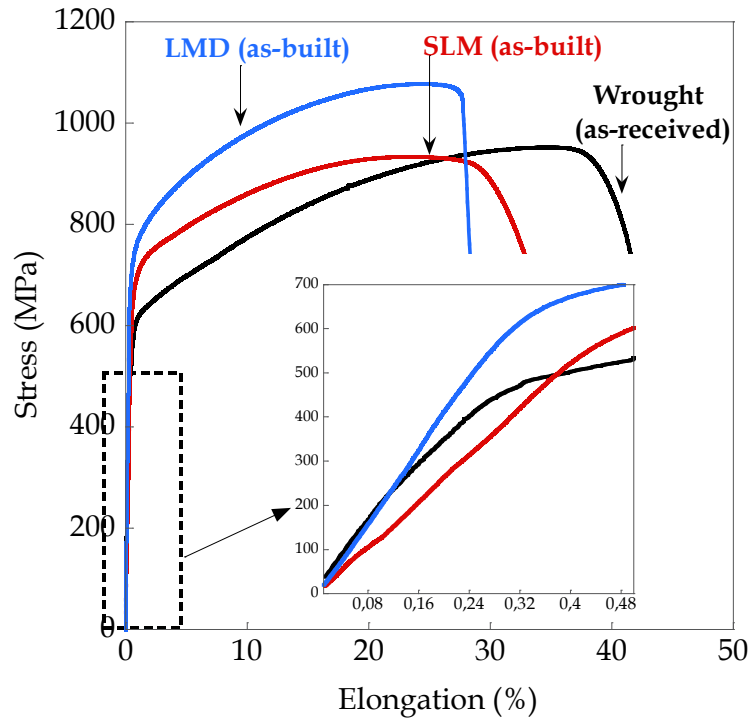


Figure 16: Tensile curves of Inconel 625. Comparison between the conventional and the additive manufacturing materials.

472 Concerning the AM specimens, the stress-strain tensile curves are shown in  
 473 Figure 16. All the specimens were tested in the laser scan direction. A compari-  
 474 son is established between the as-built AM specimens and the as-received wrought

material (commercial Inconel 625). Results show that the highest strength characteristics are obtained for the LMD specimens, with a YS 50% higher and an UTS 12% higher than those of the wrought specimens. The SLM specimens present lower strength characteristics than those of the LMD (YS and UTS) but with a YS still 35% higher than the wrought material one for an even UTS. Strong differences occur for the elongation to failure with SLM samples with a lower ductility than wrought material (22% mean drop) but higher than LMD specimens (37% drop on average compared to wrought material) (see Table 6) .

Inconel 625	Young Modulus (GPa)	Yield stress (MPa)	Ultimate tensile strength (MPa)	Elongation (%)
Wrought samples	184 ± 16	482 ± 42	955 ± 6	41 ± 1
<i>Samples processed by SLM</i>				
As-built (AS)	145 ± 4	652 ± 10	925 ± 13	32 ± 3
AS + 900°C/1 h	142 ± 11	567 ± 15	869 ± 7	38 ± 1
AS + 1100°C/1 h	114 ± 8	409 ± 14	886 ± 11	56 ± 5
<i>Samples processed by LMD</i>				
As-built (AS)	223 ± 24	723 ± 23	1073 ± 5	26 ± 2
AS + 900°C/1 h	224 ± 19	654 ± 15	1084 ± 2	27 ± 2
AS + 1100°C/1 h	213 ± 22	532 ± 22	991 ± 13	43 ± 1
<i>Other investigations</i>				
LMD [21]	-	656	1000	24
SLM [16]	182	783	1041	33
SLM [22]	196 ± 12	641 ± 24	879 ± 2	30 ± 2

Table 6: Tensile properties of Inconel 625

The inset in 16 shows a magnification of the tensile curves between 0 and 0.5% of mechanical strain. It is interesting here to compare the elastic behavior of AM and wrought specimens. Results indicate a lower Young modulus for the specimens printed by the SLM process. Moreover, the elastic evolution of LMD and wrought specimens seems more similar. These observations suggest that mechanical properties for the as-built SLM and LMD specimens are different. This may be certainly explained by the strong differences in terms of microstructure or by the dislocations structures, resulting from difference in the process. Concerning the microstructure, the EBSD analysis of the SLM specimens indeed shows a very strong texture, with a preferential orientation  $\langle 001 \rangle$ , as for a single crystal



material. In this case, the low slope of the stress-strain curve indicates that the activation of the sliding systems for plasticity proper to the cubic faced-centered (cfc) structure would be faster in the as-built SLM specimens : this hypothesis could be tested by modifying the printing strategy of SLM specimens, which would be a study in itself.

Table 6 also summarizes the literature results of tensile tests realized on horizontal Inconel 625 processed by additive manufacturing. For the as-built LMD sample, obtained tensile properties are practically similar to the literature investigation [21]. Moreover, the authors also obtained a mixed dendritic and columnar microstructure with cellular and elongated grains, in line with our observations. For the SLM samples, the elongation to failure was found closed to the literature observations [16, 22]. Nonetheless, Marchese et al.[16] determined higher tensile strengths, which can be related to the less-pronounced grains texture. Once again, the preferred  $\langle 001 \rangle$  grains orientation presented in this paper seems to explain the low value of the Young modulus and yield stress of the as-built SLM sample.

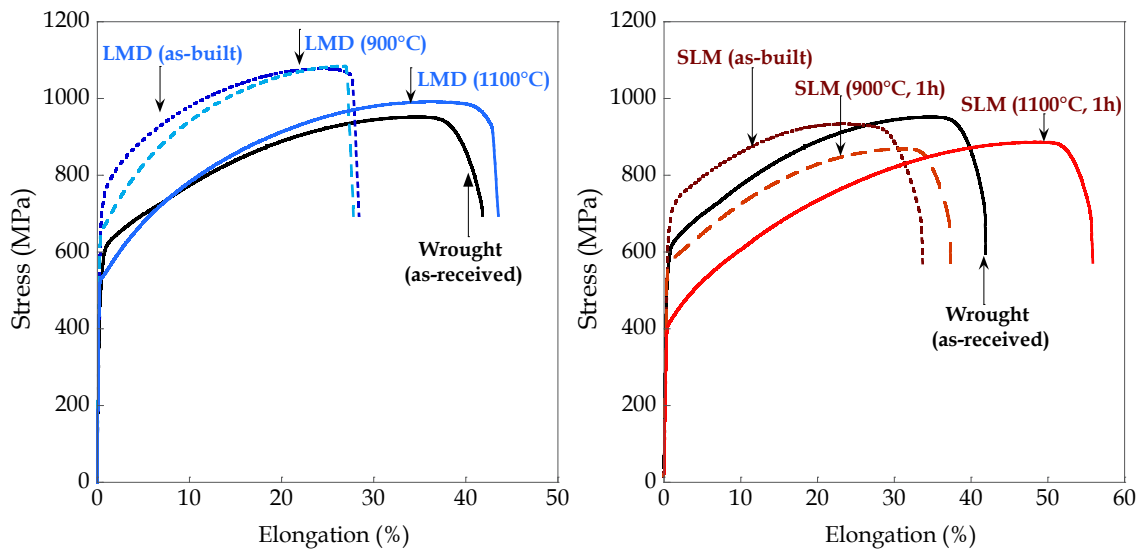


Figure 17: Effect of heat treatments on tensile properties of Inconel 625 for the LMD samples (in blue); for the SLM samples (in red).

After heat treatment, the YS and UTS for the LMD specimens are still higher than those of the wrought sample (see Figure 17.a). Compared to the as-built

LMD specimens, there is no significant change on the ultimate strength and the elongation to failure after the annealing at 900°C (Table 6). However, YS decreases slightly, probably due to the beginning of the residual stresses relaxation during the post-annealing treatment. After annealing at 1100°C, the mechanical strengths of the LMD specimens decrease again, while the elongation to failure increases. As shown in Table 6, the mechanical properties of the LMD samples heat-treated at 1100°C become similar to those of the conventional wrought material. Moreover, the obtained UTS and elongation values turn out to be slightly higher. This heat treatment favors the improvement of the ductility that increases by up to 65% compared to the as-built state and provide a material which is close to the wrought one.

523

On the other hand, for SLM specimens, heat treatment at 900°C greatly affects the tensile properties. YS decreases by 13% compared to the as-built state, while the elongation to failure increases by 19%. As shown in Table 6, the annealing at 1100°C allows here again an improvement of ductility, which increases by 75% compared to the as-built material and reaches 43%. The mechanical strengths of the SLM specimens annealed at 1100°C however become lower than the conventional wrought Inconel 625. Here, a more in-depth investigation should be carried out to find the most suitable heat treatment to obtain interesting properties for the SLM material. It is thus important to note that simple exposure to high temperatures is not sufficient enough to improve SLM material properties.

534

It is finally interesting to stress out the effect of texture and the influence of the loading direction. Elongated grains in the preferred  $\langle 001 \rangle$  orientation for SLM specimens are here perpendicular to the mechanical stress direction, which favors a faster activation of the micro-plasticity sites and therefore leads to a decrease for the mechanical resistance compared to LMD specimens. Such texture effect has been previously pointed out by Ma et al. [23]. The apparent observed ductility for printed specimens are lower for example essentially because the failure will come faster partially because of the strong texture effect. Nevertheless, if the direction of loading is parallel to the grain orientation, then the tensile properties should be better; Kreitchberg et al [17] indeed shows that the ductility for Inconel 625 specimens obtained by LMD is better when they are printed vertically than horizontally. For LMD samples, the more homogeneous texture surely makes the material less sensitive to loading direction. Once again, further investigations on texture and precipitates effect on mechanical properties have to be performed.

#### 549 4. Some discussions

550 The analysis of the previous results underlines that the high cooling rate which  
551 occurs during the additive manufacturing processes affects the grain morphology  
552 and the texture of printed parts. It is shown that the grains usually appear more  
553 elongated in the direction parallel to the build direction. A dendritic growth was  
554 therefore identified from the bottom to the top part of the melt pool. As shown  
555 previously, the bottom part of the layer mostly consists of columnar structure with  
556 cell-form dendrites, while the top part of the layer shows a very fine dendritic  
557 structure. This is a consequence of the downward progressive solidification ve-  
558 locity, which is higher at the bottom zone than at the upper one. The main pa-  
559 rameters piloting the LMD manufacturing process have thus a substantial impact  
560 on the size and the morphology of the grains. A high laser power contributes to  
561 a significant formation of elongated grains, which also appear bigger, whilst the  
562 number of cell-form dendrites decrease. Indeed, a part of the laser power may be  
563 absorbed by the powder and used to bring it to temperature and generate melt pool.  
564 The laser-deposited layer is then subjected to a non-isothermal annealing (without  
565 temperature maintain) which greatly influences the phase transformations and the  
566 recrystallization processes during the manufacturing phases. Thus, the average  
567 temperature of the laser-deposited layer is going to decrease gradually, depend-  
568 ing on its vicinity to the substrate. The farther the layer from the substrate, the  
569 lower the cooling rate [17]. Thereby, an increase in the laser power would result  
570 in a greater heat flux that may promote the formation of a hugely dendritic mi-  
571 crostructure with elongated grains.

572  
573 To go a bit further, this global "macroscopic" influence of this laser power  
574 is finally briefly investigated. Figure 18 illustrates the correlation between this  
575 manufacturing process parameter and both the microstructure and the mechani-  
576 cal properties of parts printed by LMD. For each of the parameters studied, the  
577 material with the highest value is given a maximum score of 1, so that the score  
578 for the other materials is deduced by a simple rule of three from this reference.  
579 The effect of laser power on the microstructure (ratio of elongated grains) is here  
580 obvious and highlighted by the increase of the dendritic structure with power. It  
581 is however less significant on the hardness and the yield stress. The maximum  
582 hardness is obtained for the printed part at 270W :  $343 \pm 9$  HV, while the mini-  
583 mum hardness is  $315 \pm 14$  HV, for a 300W laser power. For the yield stress, the  
584 maximum value was also obtained for the sample printed at 270W (746 MPa),  
585 and similar values were found for 300W and 330W. Therefore, it seems that the

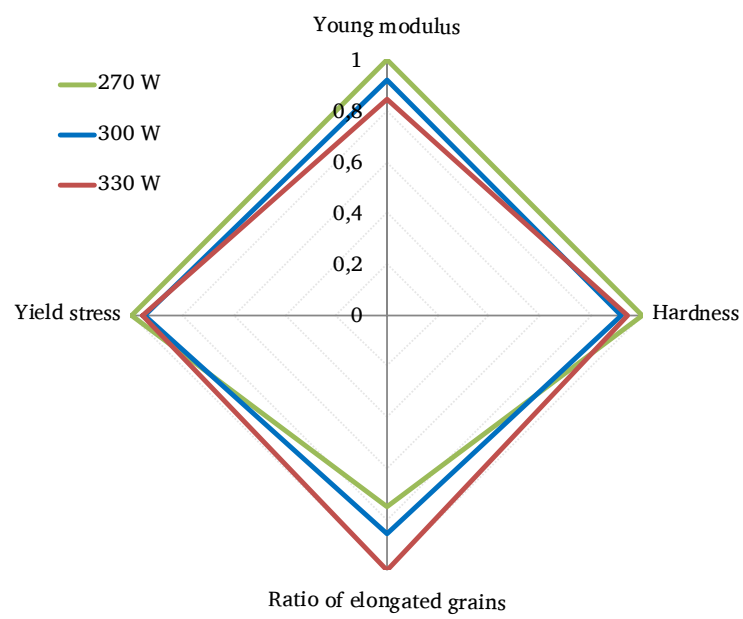


Figure 18: Correlation between the laser power of the manufacturing process, the microstructure and the mechanical properties of Inconel 625 obtained by LMD

586 elongated grains morphology does not play a significant part on the improvement  
 587 of the mechanical properties for horizontally-printed bars.

588  
 589 Finally, the Young modulus also shows its maximum value (239 GPa) at the  
 590 lowest laser power. with significant variations with a decrease of nearly 20% at  
 591 330W. These results thus emphasize a significant effect of the laser power on the  
 592 microstructure, among others, on the morphology and orientation of the grains  
 593 which in turn lightly affects the mechanical properties of the LMD printed parts  
 594 (for example, no variation on the elongation to failure was detected for samples  
 595 printed at 270, 300 and 300W).

596

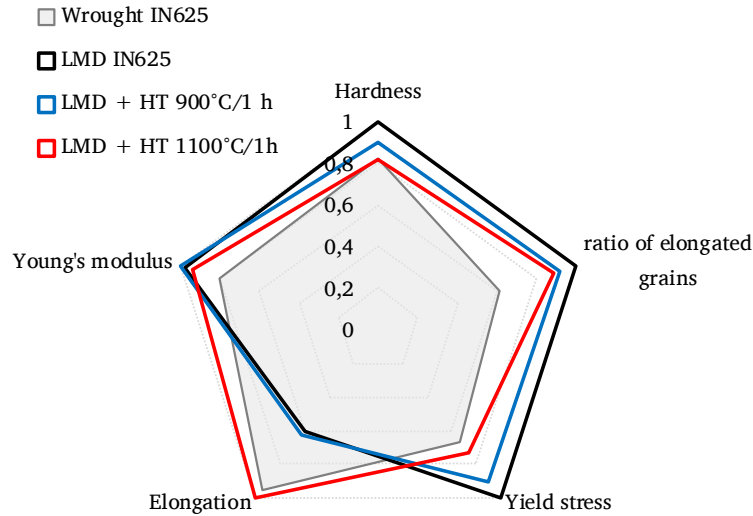


Figure 19: Effect of post-annealing treatments on the microstructure and mechanical properties of LMD samples.

597 Figure 19 finally shows the impact of the post-annealing treatment on the mi-  
 598 crostructure and mechanical properties of samples processed by the LMD. Com-  
 599 paring to the conventional wrought material, one can see that the heat-treatment  
 600 performed at 1100°C contributes to reduce the high value for hardness obtained  
 601 for the LMD as-built material, due to residual stresses. After 1h at this temper-  
 602 ature, it tends to be similar to the wrought material one. In the same way, this  
 603 post-treatment improve the grains morphology by the recrystallization process to

604 achieve an equiaxed grains microstructure close to the traditional material one.  
605 The duration of the heat-treatment at  $1100^{\circ}\text{C}$  seems however to be rather low to  
606 fully homogenize the size and the grains shape. One can also see that the strengths  
607 and the Young modulus of the LMD samples are much higher than the conven-  
608 tional wrought, even after the heat treatment. As a result, the post-annealing seems  
609 to be the most effective way to greatly improve the ductility of AM parts, while  
610 keeping good tensile strengths and finally obtain an improved printed material if  
611 compared with the wrought one.

## 612 **5. Conclusion**

613 The microstructure characteristics as well as the mechanical properties of an  
614 Inconel 625 alloy obtained by three processes: forging, SLM and LMD, were  
615 studied. For the last two processes, known as "additive manufacturing", the in-  
616 fluence of printing parameters was considered as well as the role of possible heat  
617 treatments. The main conclusions of this study are :

- 618 • Additive Manufacturing processes induce the presence of columnar den-  
619 drites with a very heterogeneous grain size for as-built materials.  
620
- 621 • The microstructures appear highly textured with a privileged grain orienta-  
622 tion, particularly for SLM: the material is thus very far from the equiaxed  
623 microstructure of the wrought alloy.  
624
- 625 • The microstructure obtained by SLM is often finer than the one obtained by  
626 LMD even if a reduction in laser power tends to refine it.  
627
- 628 • Heat treatments and particularly the 1h- $1100^{\circ}\text{C}$  annealing enable to improve  
629 the printed parts microstructure by suppressing the columnar dendrites  
630
- 631 • The hardness and tensile properties of the printed parts (yield stress and ten-  
632 sile strenght) are most of the time better than that of wrought parts but their  
633 ductility is often low.  
634

- 635     • Annealing at 1100°C improves the ductility: all the mechanical properties  
636       of the LMD material become closer to those of the wrought one.
- 637     • The LMD process with controlled laser power, coupled with appropriate  
638       heat treatment, produces both microstructures and mechanical properties  
639       close to or better than those of the wrought alloy.

640       Finally, some complementary investigations could be done in order to improve  
641       the microstructural characteristics of the as-built Inconel 625, by looking for a  
642       series of adequate parameters to achieve a more homogeneous behavior for printed  
643       material. The study of the fatigue strength of this type of printed alloy can also be  
644       an interesting property to investigate.

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651       XRD.

## 652   **7. Data availability**

653       The raw/processed data required to reproduce these findings cannot be shared  
654       at this time as the data also forms part of an ongoing study

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