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Summary

A batch of gas-atomized 316LN stainless steel powder was thoroughly characterized to determine chemical composition, particle size distribution, particles morphology, tap density and flowability. Effect of powder drying was studied on SLM specimens density, tensile properties and impact strength. Drying allowed to increase density from 96.9% to 99.6% and obtain mechanical properties higher than minimal value expected for forged 316LN. Fracture surfaces of tensile bars observed by SEM explained the differences measured in mechanical properties. Properties and energy densities are compared to previously published ones.

Introduction

316L stainless steel has been widely investigated for SLM [1-6]. The asymmetrical temperature gradient during cooling create microstructural heterogeneities. Fine cellular sub-grains are formed, with a large concentration and pile-up of dislocations at the cell boundaries and inside the cells. High mechanical strength, ductility and hardness are obtained and explained by the microstructure. However, no results are available for 316LN specimens by SLM. Whereas nitrogen content is lower than 0.11wt%. in 316L, it is between 0.12 and 0.22 wt%. in 316LN. In forged material, this content increases the stability of the austenite and the mechanical properties without compromising the tenacity. Mechanical data of additively manufactured 316LN are lacking to designers.
This works aims at understanding the effect of powder drying on the density and mechanical properties of 316LN SLM parts, as well as the difference of energy densities between a roller machine and doctor-blade type machines.

Material and experimental procedure

Powder material and characterization techniques

The 316LN (1.4429) powder used, of theoretical density 7.99 g/cm^3, was produced by gas atomization under nitrogen, sieved to below 45µm range and supplied by Erasteel. Two pre-conditioning of the powder were compared: first, the powder was used as-received without any drying (Batch A); then, after several re-uses and systematic sieving removal of particles higher than 70µm, powder was sieved at 20µm to remove small particles and dried at 170°C under nitrogen for 24 hours (Batch B). Sieving was done on a Retsch AS200 basic vibratory shaker.

Metallic elements contents and carbon, oxygen, nitrogen and sulfur contents were measured by the supplier, respectively, by Spark Optical Emission Spectroscopy (Spark OES) and Instrumental Gas Analysis (IGA). Particle size distribution was measured by laser diffraction on a Malvern Mastersizer 2000 granulometer in ethanol according to ISO 13320 standard. Median particle size (D_{50}), as well as D_{10} and D_{90} corresponding respectively to the fraction of the population below 10% and 90% of the cumulative frequency distribution, were extracted. Convexity and High Sensitivity (HS) Circularity, also called roughness and sphericity of the particles, were evaluated on a Malvern Morphologi G3® particle shape image analyzer. Powder morphology was analyzed by Scanning Electron Microscope (SEM) using a PHILIPS XL30 microscope. Apparent and tapped density were determined on a Quantachrome Autotap™ analyzer using 250 mL of powder after 1000 taps. Avalanche angle was measured on a Mercury Scientific Revolution® Powder analyzer using 80 mL of powder at a drum rotation of 0.6 rpm and 150 avalanches recorded.

Samples fabrication and heat treatments

The SLM machine used was a ProX200 (3D Systems, USA) equipped with a 300W fiber laser and a forward and backward counter-rotating roller. The focused spot size was ~80 µm. Laser power and scanning speed were in the range 130-180 W and 1200-1600 mm/s, respectively. Powder bed layer thickness and hatch spacing were fixed at 30 µm and 50 µm, respectively. A powder bed compaction factor of 57% was used. The scan strategy was the following: hexagonal design with 12 mm radius or edges and an overlap of 100 µm; the first layer was oriented at 45° angle versus X direction and next layer was crossed at 90° with respect to the preceding layer. 15*15*15 mm^3 samples (Figure 1a) were fabricated under argon on 316L platforms for density and IGA measurements. On the same platforms, 58*13*13 mm^3 parallelepipedic specimens were built in XY direction with 0° angle versus the roller direction (X) (Figure 1b). Half of the specimens of each batch was solution annealed at 1100°C under air for 15min and quenched in water. Among each batch, half of the specimens were machined to cylindrical tensile test specimens according to [7] (Figure 2a) and the other half to Charpy V-notch test specimens according to [8] (Figure 2b). The notches were machined parallel to the XY plane. Two to four samples were used for each condition.

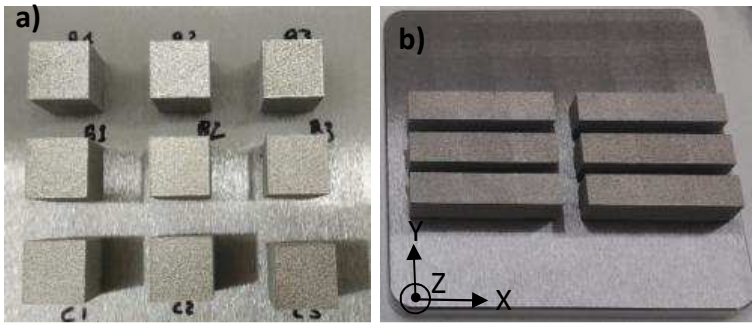

Figure 1 : a) Cubes and b) Parallelepipedic specimens after building on their platforms

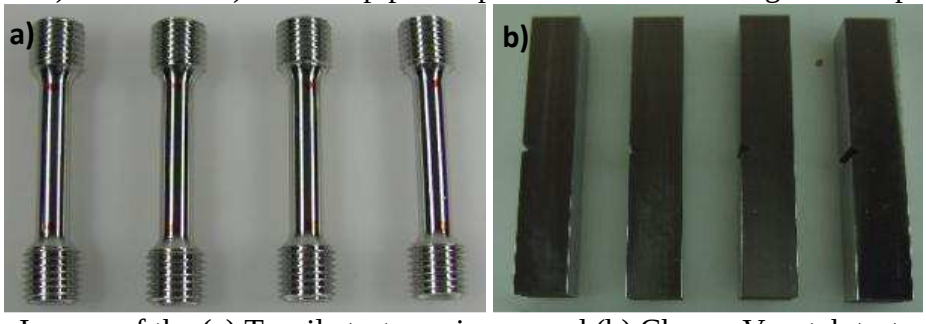

Figure 2 : Images of the (a) Tensile test specimens and (b) Charpy V-notch test specimens

Density

Density of cubes were measured by Archimedes Method using water or isopropanol. This method was preferred to image analysis of polished cross-sections, known to overestimate densities [9]. However, few samples were cross-sectioned and polished for observations under a Leica DM2500 optical microscope. Carbon, Oxygen and Nitrogen contents were measured on a SLM cube specimen by Instrumental Gas Analysis (IGA) by a sub-contractor.

Mechanical properties

Tensile tests were performed on a MTS hydraulic mechanical 100 kN test machine equipped with a clip-on extensometer ±15% with a gage length of 10 mm, at a test speed of 5.10^{-4} s^{-1} at room temperature. The ultimate tensile strength (UTS) and the 0.2% offset yield strength (YS) were determined from the stress-strain curves and elongation (A) from the measurement of the elongation of the gage section. Charpy tests were done on a Zwick/Roell impact test machine equipped with a pendulum of 300J. The fracture surfaces of the specimens were observed by Scanning Electron Microscope (SEM) using a ZEISS LEO 1500 field emission gun (FEG) microscope.

Results

Powder characterization

Results of chemical analyses of the powder supplier are compared, in Table 1 and Table 2, to the standard. Spark spectrometry results are in line with the standard for all elements. Carbon, oxygen, nitrogen and sulfur contents are also consistent with the standard.

	Fe (%pd)	Mn (%pd)	Ni (%pd)	Cr (%pd)	Mo (%pd)	Si (%pd)	P (%pd)
316LN specification [10]	Bal.	≤ 2	11-14	16.5-18.5	2.5-3	≤ 1	<0.045
Supplier analysis	Bal.	0.06	13.8	17.8	2.5	0.3	0.004

Table 1 : Metallic contents of the as-received 316LN powder

	C (%pd)	O (%pd)	N (%pd)	S (%pd)
316LN specification	≤ 0.03	< 0.5	0.12-0.22	< 0.015
Supplier analysis	0.024 ± 0.0017	NA	0.14 ± 0.006	0.006

Table 2 : Carbon, oxygen, nitrogen and sulfur contents of the as-received 316LN powder [10]

For both powders, as-received (Batch A) and "re-used and sieved" (Batch B), the particle size distribution are monomodal (Figure 3). The sieved powder still contains fines, probably because of a too low residence time in the sieve. Compared to the as-received powder, D_{10} increased only from 12µm to 14µm (Table 3); D_{50} and D_{90} are similar.

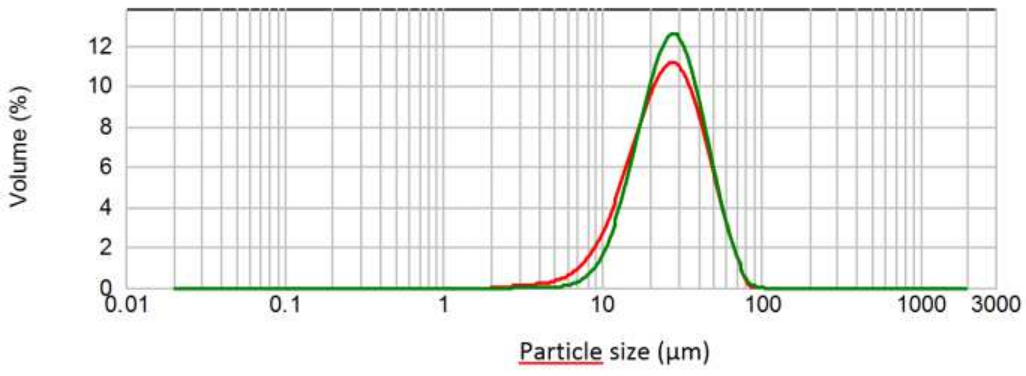

Figure 3 : Particle size distribution of 316LN powder as-received (red curve) and "re-used and sieved" (green curve)

	D_{10} (µm)	D_{50} (µm)	D_{90} (µm)
Supplier*	8.8	25.2	46.5
As-received	12.0	25.6	48.2
Re-used and sieved	14.0	26.5	47.5

* Experimental conditions unknown

Table 3 : Comparison of deciles 10, 50 and 90 on the particle size of the 316LN powder

For the as-received powder, the median HS Circularity, given by HS Circularity = $4\pi.A/P^2$ (A = surface area; P=perimeter), is 0.86 ± 0.02, which means that 50% of the volume of the powder has a sphericity higher than 0.86. When it is equal to 1, the particles are perfectly spherical. For comparison, gas-atomized powders used for SLM have HS circularities between 0.79 and 0.99. The median convexity of the as-received powder is 0.97 ± 0.01. Convexity is defined as the ratio between the perimeter of the particle envelope (Hull perimeter) and the actual perimeter of the particle. The space between the envelope of the particle and its actual perimeter is assimilated to roughness. A perfectly smooth surface particle would have a convexity of 1 whereas a strongly rough particle will show a lower convexity. For comparison, gas-atomized powders used for SLM have convexities between 0.95 and 0.995.
The particles are globally spherical and rough at the surface (Figure 4). Many of them show one or two satellites. Few granules are truncated or non-spherical, which may have an effect on powder flowability.

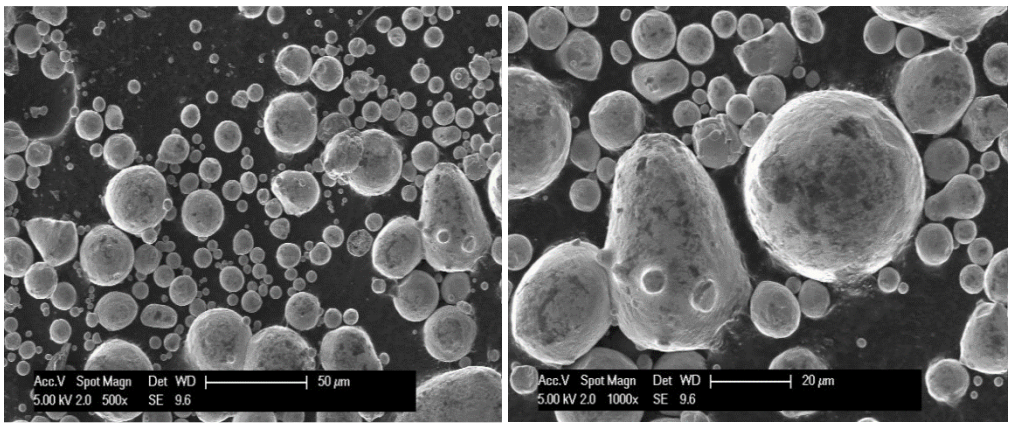

Figure 4 : SEM images at two magnifications showing the as-received 316LN powder morphology

Despite the small difference on D_{10} between as-received and sieved powder, the effect of sieving on the avalanche angle is significant, with a decrease from 55.6 ± 2.8° to 47.8 ± 2.4°. The effect would probably have been even more marked with longer sieving. The high avalanche angle of as-received powder is explained by the high proportion of fine particles and the presence of satellites. For comparison, 15-45µm gas-atomized powders used classically for SLM have avalanche angle between 32° and 45°.
Removing a part of the fines lead to a decrease in apparent density (Table 4), but tap densities of the powders are similar. Relative densities are about 65% for both powders. Those high densities of the packed powder bed are favorable to a complete densification after laser melting.

	Apparent density	Tap density (g/cm^3)	Relative density after tapping
As-received	4.77 ± 0.02 g/cm^3	5.24 ± 0.02 g/cm^3	65.6 ± 0.5 %
Re-used and sieved	4.54 ± 0.02 g/cm^3	5.25 ± 0.02 g/cm^3	65.7 ± 0.5 %

Table 4 : Apparent and tap densities of powder batches A and B

SLM cubes characterization

With un-dried powder (Batch A), the highest measured density, obtained with a laser power of 135 W and a scanning speed of 1400 mm/s, was 96.9%. Increasing the energy density did not allow to increase the specimen density. With the powder dried at 170°C under N_2 (Batch B), the highest measured density, obtained with a laser power of 170 W and a scanning speed of 1400 mm/s, was 99.6%. Comparison of polished cross-sections of samples from batch A and batch B (Figure 5) confirm visually the Archimedes results.

At a given energy density, defined by E = P / (V*h*e) where P is the laser power, V the scanning speed, h the hatch spacing and e the layer thickness [11], the preliminary drying of the powder makes it possible to achieve higher specimen densities (Figure 6).

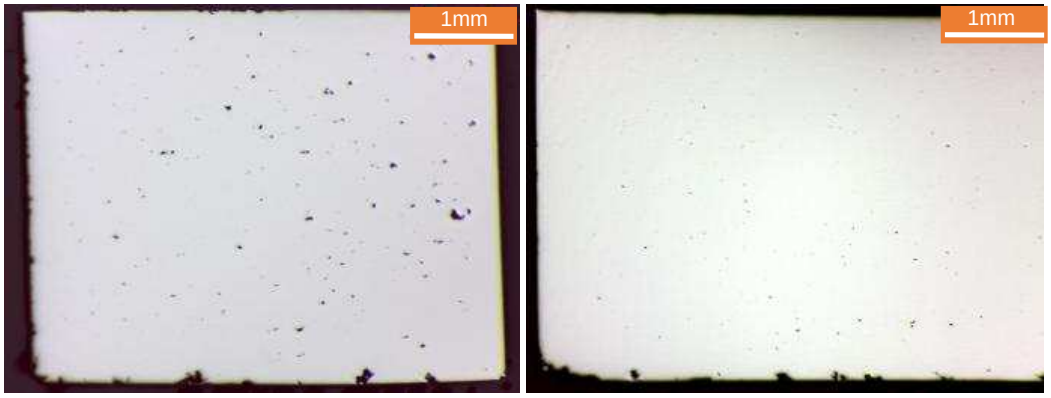

Figure 5 : Optical cross-sections of 316LN SLM samples fabricated with : a) un-dried powder and b) 170°C N_2 dried powder

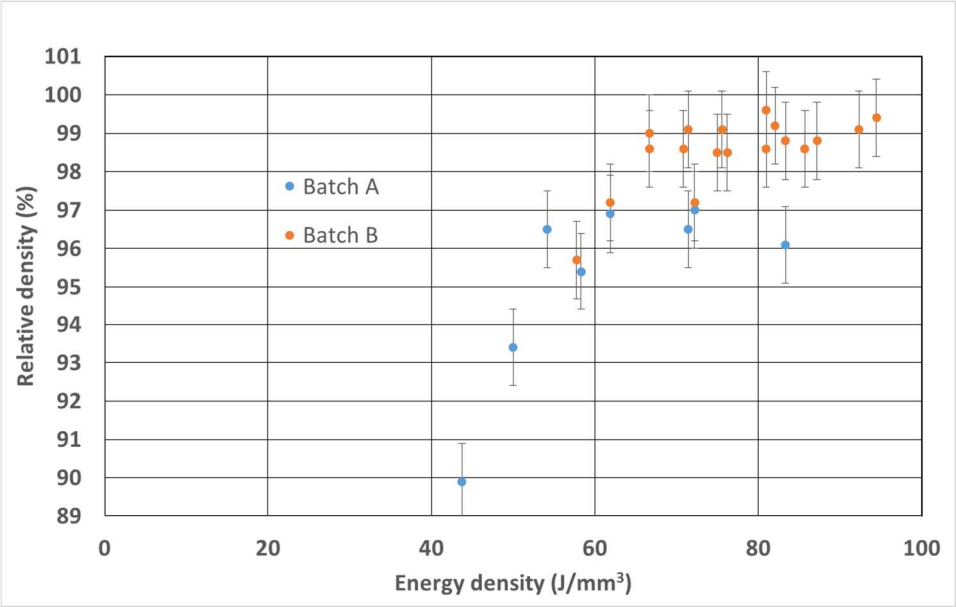

Figure 6 : Evolution of the specimen density as a function of the energy density

Density measurements were carried out on two of the cubes to evaluate the effect of the measuring fluid; the densities measured under isopropanol (IPA) are similar or higher than with water (Table 5), which confirms the results from [9]. Carbon, oxygen and nitrogen contents measured on a SLM specimen obtained from powder B are still consistent with the standard and close to the values of the powder.

Sample	Archimedes Fluid	Density (g/cm^3)	Relative density (%)
Cube 1	Water	7.94	99.42
	IPA	7.97	99.71
Cube 2	Water	7.92	99.18
	IPA	7.92	99.17

Table 5 : Effect of measuring fluid on cubic densities

	C (%pd)	O (%pd)	N (%pd)
316LN specification [10]	≤ 0.03	< 0.5	0.12-0.22
As-received powder (Supplier)	0.024 ± 0,0017	NA	0.14 ± 0,006
SLM specimen (re-used and sieved powder)	0.028 ± 0,002	0.027 ± 0,002	0.12 ± 0,01

Table 6 : IGA results of a SLM specimen compared to initial powder and standard

Mechanical properties

Densities of parallelepipedic specimens fabricated from batch A were measured by Archimedes method. While the cube density of the preliminary study was 96.9%, parallelepipedic specimens performed in the same conditions showed relative densities from 93% to 96.9%. This density variation could be explained by local moisture variations, which, in turn, could produce gases during melting; parts of these gases are trapped during solidification. Densities of specimen from batch B were measured on control cubes build on the same platform as parallelepipedic specimens; densities were 99.45% and 99.46% on as-build and solution annealed cubes, respectively.

Tensile properties and impact strength of specimen from batch B are much higher than the ones from batch A (Table 7), both in the as-received and the solution annealed states. As sieving had a limited effect on powder tap density, it is believed that drying of the powder is the main cause of this increase in mechanical properties. Properties of solution annealed specimens produced from dried powder are higher than minimal value expected for forged 316LN.

	Material grade	Building Direction & State	YS (MPa)	UTS (MPa)	A (%)	Impact strength Kv (J)
Hot-rolled and solution annealed flat product [10]	316LN (1.4429)		>280	580-780	> 40	60(tr)-100(l)
Batch A (virgin powder, not dried)	316LN	XY; machined surface	526 ± 28	611 ± 30	10 ± 3	23 ± 3 **
		XY; HT° 1100°C; machined surface	420 ± 13	623 ± 41	24 ± 5	28 ± 13 **
Batch B (re-used powder, 20-70µm sieved, dried 170°C/N2)	316LN	XY; machined surface	630 ± 4	721 ± 4	46 ± 1	92 ± 1 *
		XY; HT° 1100°C; machined surface	456 ± 3	682 ± 2	47 ± 1	102 ± 4 *

Table 7 : Tensile test and Charpy-V test results for 316LN

Fracture surfaces of tensile bars explain the differences in mechanical behavior between the two batches: samples from un-dried powder present a high density of pores with un-molten particles, both for as-build (Table 7a) and solution annealed (Table 7b) samples. On the contrary, very few pores and un-molten particles are visible on fracture surfaces from samples produced with dried powder (Figure 8).

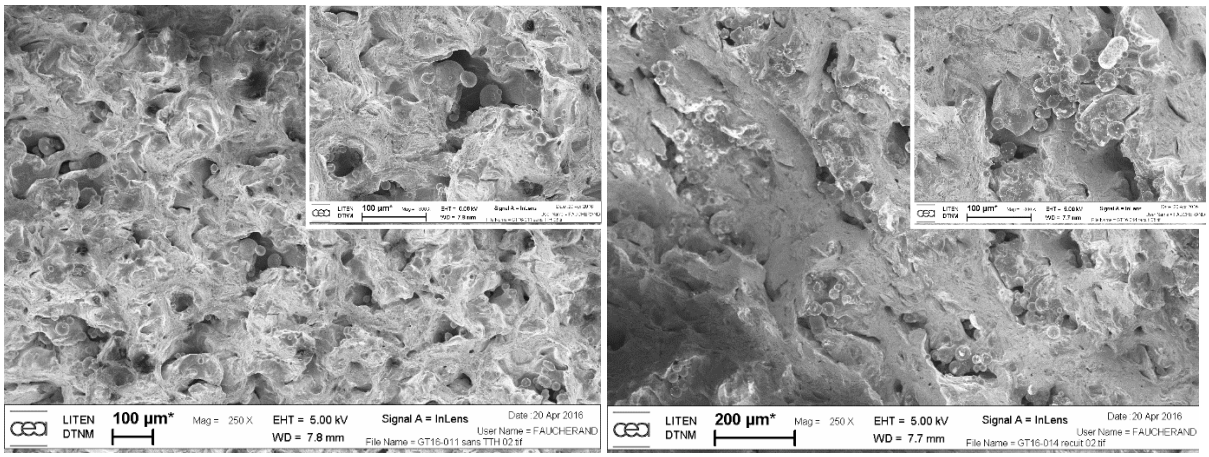

Figure 7 : a) Fracture surface of as-build tensile specimen from Batch A ; b) Fracture surface of solution annealed tensile specimen from Batch A

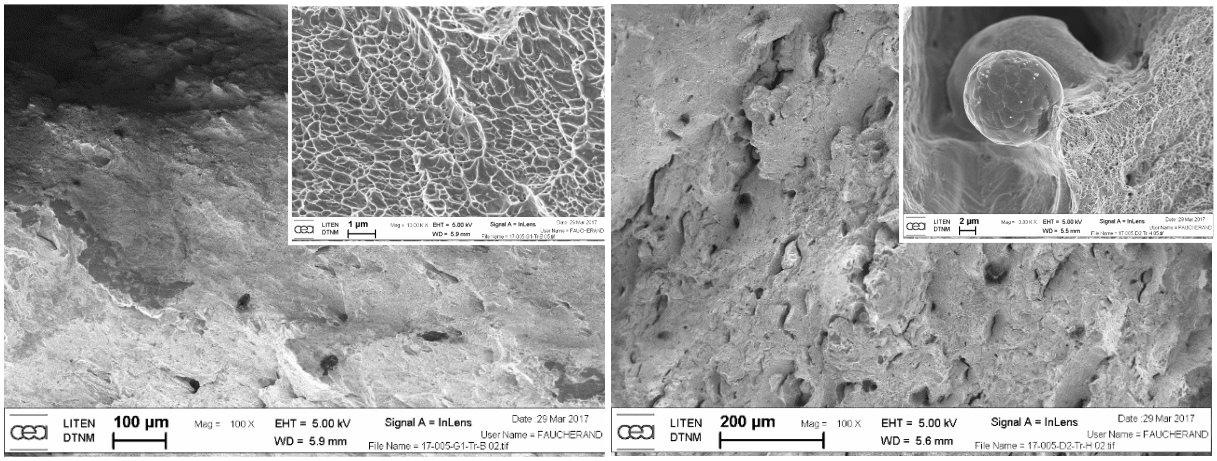

Figure 8 : a) Fracture surface of as-build tensile specimen from Batch B ; b) Fracture surface of solution annealed tensile specimen from Batch B

Discussion

In as-build state (green lines of Table 8), YS, UTS and elongation are mostly higher than published values for 316L and 304L; however, impact strength is lower than the one of [4] for 316L, despite a similar density (99.3%). In the solution annealed state, YS and UTS are higher than published values; elongation is comparable and impact strength is, again, lower than the only value published. Elongation and impact strength could be probably increased with a hot isostatic pressing post-treatment.

	Material grade	Building Direction & State	YS (MPa)	UTS (MPa)	A (%)	Impact strength Kv (J)
Batch B (re-used powder, 20-70µm sieved, dried 170°C/N2)	316LN	XY; machined surface	630 ± 4	721 ± 4	46 ± 1	92 ± 1 *
		XY; HT° 1100°C; machined surface	456 ± 3	682 ± 2	47 ± 1	102 ± 4 *
[1] (Realizer MCP AM 250; Heat treatment not mentionned)	316L	XY with 30° angle/X	678 ± 28	691 ± 34	33 ± 5	63,3 ± 6,1
[3] (SLM Solutions 280)	316L	XY; turned surface	482	636	36,6	
		XY; HT° 1080°C; turned surface	322	593	52,9	
[9] (Renishaw AM250; No heat treatment)	304L	XY	NC	689 ± 5	40	
[4] (SLM Solutions 280)	316L	XY; turned surface	500 ± 10	630 ± 12	39 ± 6	120 ± 17 *
		XY; HT° 1095°C; turned surface	370 ± 1	612 ± 3	50 ± 1	120 ± 10 *

Table 8 : Mechanical properties of 316LN SLM vs 316L/304L from literature (* horizontal notch) in as- build (green lines) and solution annealed (blue lines) states

When comparing the input energy density in this study to the ones of others publications (Table 9), we realize that our energy is 20% to 30% lower. This difference could be explained by the compacting effect of the forward and backward counter-rotating roller of the 3DSystems machine. Compared to doctor-blade machines used in cited publications, the roller increases the powder bed density, which requires less input energy for melting.

	Material	Maximal density (%)	Energy density (J/mm^3)	Machine	Laser	Spotsize (µm)	P (W)	V (mm/s)	Layer thick. (µm)	Hatching (µm)
[2]	316	98.6 (Archi)	119	Eossint M270	Fiber	70	190	800	20	100
[9]	304L	98.8 (IA)	119	Renishaw AM250	Fiber	70	200	639	50	52.5
[12]	316L	99.2 (Archi)	101	SLM 250HL	Fiber	80	380	3000	50	25
This study	316LN	99.6 (Archi)	81	3DS Prox200	Fiber	70-80	170	1400	30	50

Table 9 : Comparison of energy densities from literature used for 316L/304L fabrication on doctor-blade type machines

Conclusion

Drying of the 316LN powder largely increased specimens density, as well as tensile properties and impact strengths. Properties of solution annealed specimens produced from dried powder are higher than minimal value expected for forged 316LN. YS and UTS are higher than published values for SLM solution-annealed 316L; elongation is comparable and impact strength is lower than the only published value. These results were obtained with a 20% to 30% reduction of energy density compared to previous publications on SLM stainless steels.

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