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Physical, Metallurgical and Mechanical Characterization Of An Austenitic Stainless Steel Produced By Selective Laser Melting

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Abstract

Complex parts of Selective Laser Melted (SLM) 316L stainless steel are now proposed as standard products by additive manufacturing companies and have been thoroughly studied on blade-type machines. In this study, a 10-45µm 316L powder batch was characterized and used to produce specimens on a forward and backward counter-rotating roller machine. Effects of solution annealing on density and microstructure of SLM cubes were studied. Tensile properties, impact strength and hardness of as-built and heat-treated samples were compared to forged materials and data of 316L SLM produced on blade-type machines.

Introduction

316L stainless steel has been widely investigated for SLM [1-6]. The sharp 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 316L specimens produced on forward and backward counter-rotating roller SLM machine, which allow high powder bed densities. This works aims at understanding the effect of the heat treatments on the microstructural and mechanical behavior of 316L SLM parts and comparing the mechanical properties to the ones of SS produced on doctor-blade type machines.

Material and experimental procedure

Powder material and characterization techniques

The 316L (1.4404) powder used, of theoretical density 7.99 g/cm³, was produced by gas atomization under nitrogen, sieved to 10-45µm range and supplied by PRS Concept. The powder's chemical composition was analyzed by two techniques: Metallic elements contents were measured on two powder samples by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) on an Agilent 700 spectrometer. Carbon, Oxygen and Nitrogen contents were measured on three powder samples by Instrumental Gas Analysis (IGA) by a sub-contractor according, respectively, to standards ISO 15350, ISO15351 and EN10276. True density was measured by He pycnometry on a Micromeritics AccuPyc II 1340 Pycnometer. Particle size distribution was measured by laser diffraction on a Malvern Mastersizer 2000 granulometer in ethanol according to ISO 13320 standard. Median particle size (D₅₀), as well as D₁₀ and D₉₀ corresponding respectively to the fraction of the population below 10% and 90% of the cumulative frequency distribution, were extracted. Aspect ratio was evaluated on a Malvern Morphologi G3® particle shape image analyzer using x20 magnification. Powder morphology was analyzed by Scanning Electron Microscope (SEM) using a ZEISS LEO 1500 field emission gun (FEG) microscope. Particle cross-section prepared by epoxy resin embedding and mechanical polishing were observed by SEM and qualitative chemical analyses were performed by Energy-Dispersive X-Ray Spectroscopy (EDX). 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 in this work 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, scanning speed, powder bed layer thickness and hatch spacing were fixed at 160 W, 1300 mm/s, 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. 13*13*13 mm³ samples for density measurements and microstructural analyses were fabricated under argon on 316L platforms. Half of them were solution annealed in a Pyrox oven at 1100°C under air for 15min and water cooled. On the same platforms, 58*13*13 mm³

parallelepipedic specimens were built in two directions (i) XY with 0° angle versus the roller direction (X) and (ii) Z (Figure 1). For each orientation, one third was heat treated at 950°C under air for 15min and another third solution annealed at 1100°C under air for 15min. Both batches were then quenched in water. As-build samples were used as reference batch. 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. Three to four samples were used for each condition.

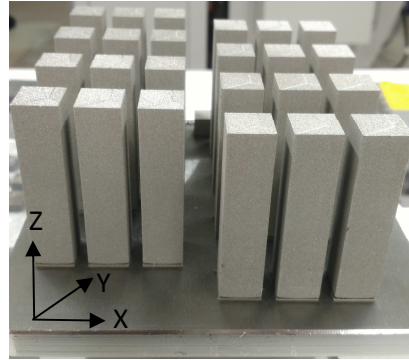


Figure 1 : Parallelepipedic specimens after building

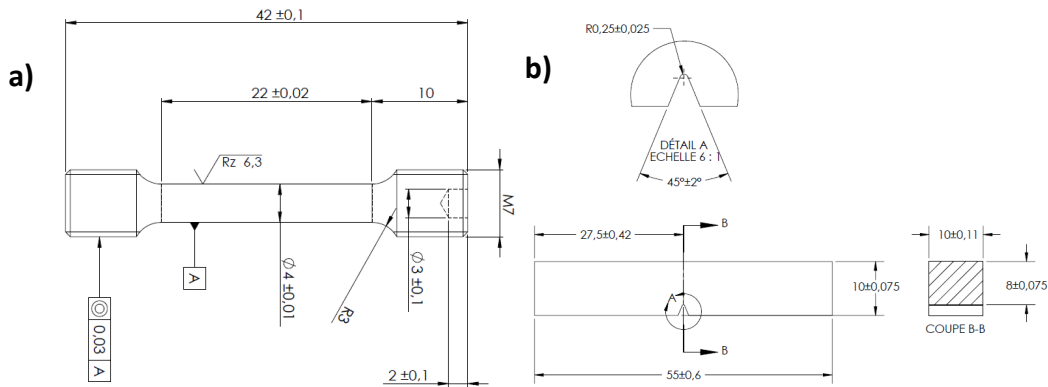


Figure 2 : Dimensions of the (a) Tensile test specimens and (b) Charpy V-notch test specimens

Density and microstructure

Density of cubes were measured by Archimedes Method using isopropanol. This method was preferred to image analysis of polished cross-sections, known to overestimate densities [11]. SEM observations and Electron Back-Scattered Diffraction (EBSD) measurements were carried out in a ZEISS Merlin FEG-SEM equipped with a Bruker EBSD system. For SEM and EBSD studies, the samples were cut parallel to the building direction, mechanically polished with standard procedures and a final polishing step was applied for few hours on a Vibromet (Buehler) table with colloidal silica suspension. Transmission Electron Microscope (TEM) observations were performed using a FEI-OSIRIS microscope. TEM samples were prepared using a twin-jet electropolishing at 20°C under 17 V with an electrolyte of 10% perchloric acid and 90% ethylene glycol monoethyl ether.

Mechanical properties

Tensile tests were performed on a MTS hydraulic mechanical 100 kN test machine equipped with a clip-on extensometer $\pm 15\%$ with a gage length of 10 mm, at a test speed of $5 \cdot 10^{-4} \text{ s}^{-1}$ at room temperature according to [7]. 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 according to [8]. A Matsuzawa MMT-X7B tester was used for measuring the Vickers micro-hardness with 1 kg load for 5 s with ten repetitions per sample.

Results

Powder characterization

Results of chemical analyses in Table 1 are compared to the standard and the supplier analyses. ICP results are in line with the standard except for the chromium content which is a bit too low. Regarding

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IGA results, carbon, oxygen and nitrogen contents are consistent with supplier ones. Taking into account the measurement uncertainty, the nitrogen content is at the upper limit of 316L according to EN 10028-7 standard. For the following, we consider that our material is a 316L grade.

	C (wt%)	O (wt%)	N (wt%)	Cr (wt%)	Fe(wt%)	Mn(wt%)	Mo(wt%)	Ni(wt%)	P(wt%)	S(wt%)	Si(wt%)
316L [9]	≤0.03	<0.5	≤0.10	16.5-18.5	Bal.	≤2	2-2.5	10-13	≤0.045	≤0.015	≤1
Supplier	<0.01	0.05	0.11	16.88	Bal.	1.03	2.48	11.77	0.04	<0.01	0.31
ICP-OES	-	-	-	15.78 ±0.74	Bal.	1.04 ±0.00	2.48 ±0.03	11.58 ±0.42	-	-	0.27 ±0.02
IGA	0.008 ±0.001	0.058 ±0.003	0.11 ±0.01	-	-	-	-	-	-	-	-

Table 1 : Composition of 316L powder

The true density of the as-received powder measured by He pycnometry is 7.994 g/cm³; after drying at 60°C, it increases to 8.014 g/cm³. In both cases, the density is slightly higher than the theoretical density of 316L (7.99 g/cm³), which suggests that the particles have little or no occlusal porosity. The particle size distribution is monomodal and narrow (Figure 3), with D₁₀ = 18.1 ± 0.9 μm, D₅₀ = 27.7 ± 0,8 μm and D₉₀ = 41.8 ± 2,1 μm. As expected, the powder does not contain particles below 10 μm. An apparent density of 4.50 ± 0.02 g/cm³ and a tap density of 4.79 ± 0.02 g/cm³ are determined. The relative density after tapping is 59.5 %. The powder shows a good flowability with an avalanche angle of 39.9° ± 2.0°.

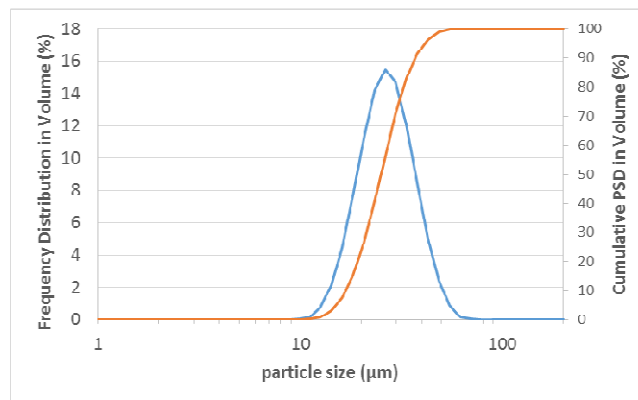


Figure 3 : Particle size distribution of 316L powder

The mean aspect ratio of the powder, i.e. the ratio between the width and the length of a particle, is 0.85 and comparable to other SLM powders. It would be 1 for perfectly spherical particle. The powder morphology characterization by SEM shows spherical particles with few satellites and misshapen particles (Figure 4).

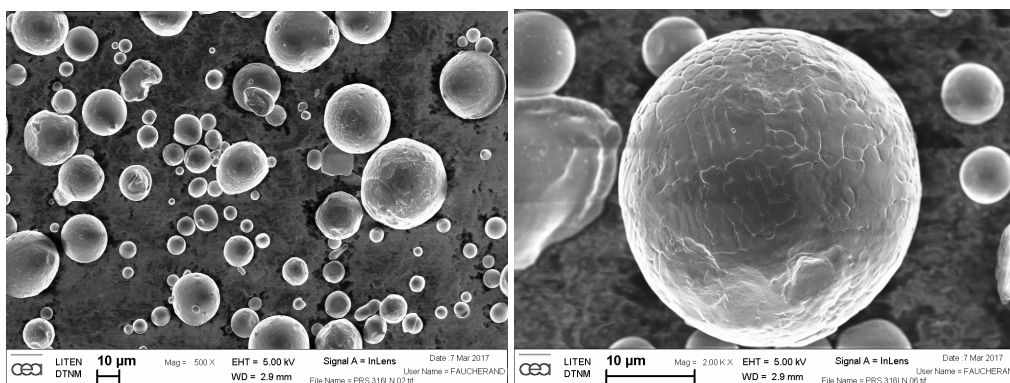


Figure 4 : SEM images at two magnifications showing the 316L powder morphology

Observation of a polished cross-section indicates that it is polycrystalline with few small porosities (Figure 5a). Back-scattering images reveals a chemical contrast at grain boundaries, which is identified as a molybdenum enrichment by EDX analyses (Figure 5b).

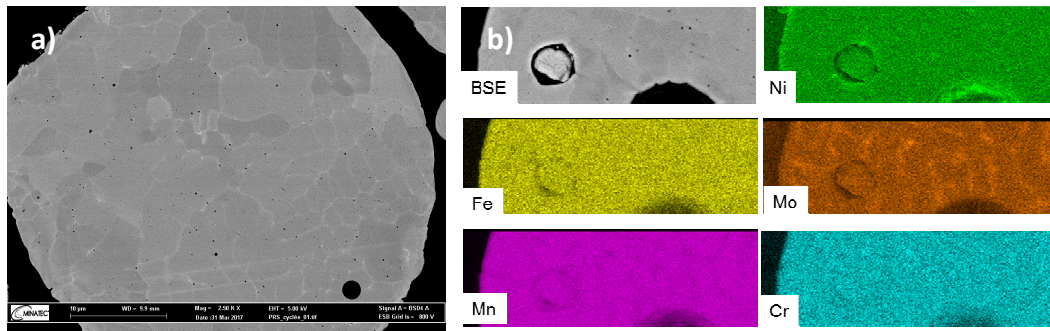


Figure 5 : a) SEM cross-section of a particle; b) EDX analysis showing Mo-rich grain boundaries

SLM cubes characterization

The highest density measured is 99.5%. Density of as-build and annealed samples were measured (Table 2). SEM observations show columnar grains grown parallel to the building direction and several porosities of few microns. The heat treatment has low impact on the microstructure; a small restauration is noticed but no recrystallization (Figure 6). EBSD analysis on as-build samples (Figure 7a) reveals a 100% austenitic microstructure, a high <100> texture along the building direction and the presence of a large number of sub-grains boundaries, which result from the formation of dislocations walls. In several sub-grains, a continuous rotating gradient is observed; it corresponds to un-organized dislocations distribution, which means high internal stresses. After annealing (Figure 7b), the material still contains a large number of sub-grains boundaries, but the continuous rotation gradient is nearly not observed, which indicates a decrease in internal stresses. Mean grain size is the same for as-build and heat treated samples, around 50-60 μm.

	Density (g/cm ³)	Relative density (%)
As-build	7.92 ± 0.01	99.19 ± 0.12
After 1100°C heat treatment	7.93 ± 0.03	99.22 ± 0.40

Table 2 : Density values for as-build and heat treated 316L samples

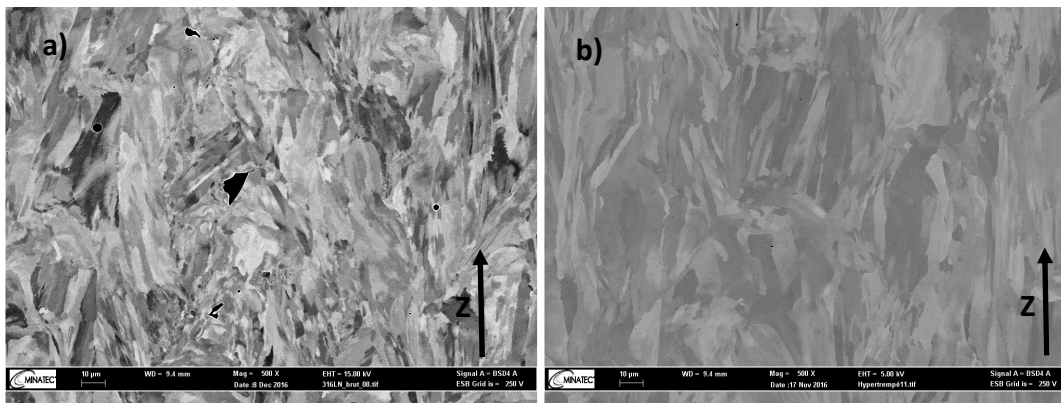


Figure 6 : SEM vertical cross-sections of 316L samples: a) As build; b) After heat treatment

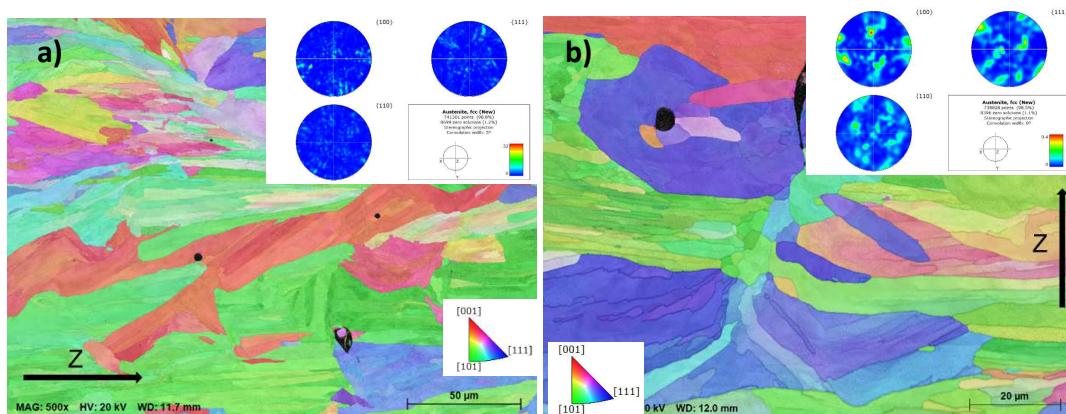


Figure 7 : IPF maps obtained by EBSD plotted with respect to the building direction (Z) for: a) As-build sample; b) 1100°C heat treated sample

TEM observations (Figure 8) reveal, in as-build sample (Figure 8a), a large number of sub-grains boundaries (indicated by solid lines) containing a large density of dislocations organized in dislocation cells (indicated by dashed lines). This high dislocation density is probably the result of the high internal stresses introduced during the process. The sub-grains (1 or 2 μm width) observed by TEM are consistent with the sub-grains detected by EBSD. The heat treatment leads to dislocation cell disappearance within the sub-grains boundaries (Figure 8b) but the sub-grains boundaries do not evolve. This dislocation restoration process lowers the dislocation density and probably relaxes a part of the internal stresses. However, dislocations density remains quite high.

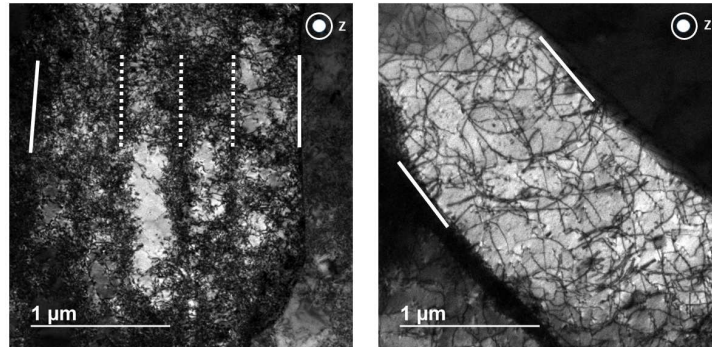


Figure 8 : TEM images of 316L samples: a) As-build; b) After heat treatment (sub-grains boundaries are indicated by solid lines whereas dislocations cells are indicated by dashed lines)

Mechanical properties

The tensile and Charpy-V results in as-build, 950°C-heat treated and 1100°C-heat treated conditions are shown for XY and Z orientations in Table 3. Values for forged water-quenched 316L and 316LN SS according to [9] are given for comparison. First, the excellent properties are the sign of the high density of the material. As observed for other SLM-materials, yield strength (YS), ultimate tensile strength (UTS) and elongation (A) values are lower for the samples build parallel to the building direction compared to the samples build in the XY plane. Impact strengths are close. The 950°C heat treatment reduced both YS and UTS with a higher effect on the former; this decrease is explained by the reduction of the dislocations density. Elongation and impact strength remain within the same order of magnitude as for as-build samples. If chromium carbides had precipitated, YS and ductility would have, respectively, increased and decreased. TEM analyses on 950°C samples would allow validating this hypothesis. The 1100°C annealing reduces further the YS and UTS, but has little effect on elongation and impact strength. All “1100°C samples”, for both directions, exhibit much higher YS compared to the standard, even compared to 316LN, and UTS values within its limitations. Elongation is also over the minimal expected values for forged 316LN. Impact strengths are largely over the minimal value expected for forged material tested in the transverse direction and slightly higher than the value in the longitudinal direction. Vickers hardness results (Table 4) are compared to values from two studies on 316L SLM. Value for forged 316L and 316LN from [10] are also shown. In as-build conditions, our values are close to the ones of [4] but largely lower than the one of [2], whose material includes silica nano-particles known for their hardening effect. As expected, hardness is reduced after heat treatment due to the reduction of the dislocations density. It is below the standard limit for forged 316L and 316LN.

	Material grade	Building Direction & State	YS (MPa)	UTS (MPa)	A (%)	Impact strength Kv (J)
	316L (1.4404)		>220	520-670	>45	60(tr)-100(l)
	316LN (1.4406)		>280	580-780	> 40	60(tr)-100(l)
This study (3D Systems ProX200)	316L	XY; turned surface	646 ± 12	753 ± 4	55 ± 4	113 ± 1 **
		Z; turned surface	551 ± 1	652 ± 2	46 ± 1	114 ± 17 **
		XY; HT° 950°C; turned surface	463 ± 4	698 ± 3	56 ± 3	116 ± 30 **
		Z; HT° 950°C; turned surface	403 ± 15	610 ± 22	54 ± 1	143 ± 33 **
		XY; HT° 1100°C; turned surface	413 ± 17	665 ± 24	45 ± 7	102 ± 2 **
		Z; HT° 1100°C; turned surface	378 ± 5	593 ± 12	54 ± 10	118 ± 13 **

** Notch parallel to the XY plane

Table 3 : Tensile test and Charpy-V test results for 316L

	This study (316L; XZ plane)	[2] (316L)	[4] (316L)	[10] Forged 316L	[10] Forged 316LN
As-build	237 ± 3,4	325	245 ± 21		
Solution annealed	193,9 ± 3,7	190	212 ± 20	217 max	217 max

Table 4 : Hardness in as-build and solution annealed states compared to literature

Discussion

As build, whatever the orientation, YS, UTS and elongation are mostly higher than the ones of 316L SLM or 304L SLM already published (Table 5). Measured impact strength is of the same order of magnitude than the one published by [4] for 316L. In solution-annealed state, whatever the orientation, YS and UTS are higher than the ones of 316L SLM already published (Table 6). Elongations and impact strengths are similar.

	Material grade	Building Direction & State	Specimens	YS (MPa)	UTS (MPa)	A (%)	Impact strength Kv (J)
This study (3D Systems ProX200)	316L	XY; turned surface	Cylindrical Ø4mm	646 ± 12	753 ± 4	55 ± 4	113 ± 1 **
		Z; turned surface		551 ± 1	652 ± 2	46 ± 1	114 ± 17 **
[1] (Realizer MCP AM 250; Heat treatment not mentioned)	316L	XY with 30° angle/X	ASTM (flat)	678 ± 28	691 ± 34	33 ± 5	63,3 ± 6,1
		Z with 0° angle/horizontal		557 ± 14	591 ± 12	42 ± 2	50,3 ± 12,7
[3] (SLM Solutions 280)	316L	XY; turned surface	NC	482	636	36,6	
		Z; turned surface		439	546	31	
[11] (Renishaw AM250; No heat treatment)	304L	XY	ASTM E8	NC	689 ± 5	40	
		Z		NC	551 ± 14	43	
[5] (SLM Solutions 250HL)	316L	Z; surface as build; as build	ISO6892-	462 ± 5	565 ± 5	53,7 ± 3	
[4] (SLM Solutions 280)	316L	XY; turned surface	ASTM E8M & E23	500 ± 10	630 ± 12	39 ± 6	120 ± 17 *
		XZ; turned surface		500 ± 21	624 ± 20	47 ± 4	128 ± 17 **

Table 5 : Mechanical properties of as-build 316L SLM vs as-build 316L/304L from literature

	Material grade	Building Direction & State	Specimens	YS (MPa)	UTS (MPa)	A (%)	Impact strength Kv (J)
This study (3D Systems ProX200)	316L	XY; HT° 1100°C; turned surface	Cylindrical Ø4mm	413 ± 17	665 ± 24	45 ± 7	102 ± 2 **
		Z; HT° 1100°C; turned surface		378 ± 5	593 ± 12	54 ± 10	118 ± 13 **
[3] (SLM Solutions 280)	316L	XY; HT° 1080°C; turned surface	NC	322	593	52,9	
		Z; HT° 1080°C; turned surface		307	553	50,7	
[4] (SLM Solutions 280)	316L	XY; HT° 1095°C; turned surface	ASTM E8M & E23	370 ± 1	612 ± 3	50 ± 1	120 ± 10 *
		XZ; HT° 1095°C; turned surface		360 ± 7	597 ± 11	35 ± 4	102 ± 13 **

Table 6 : Mechanical properties of solution annealed 316L compared to literature

Conclusion

Solution annealing at 1100°C shows little grain enlargement compared to the as-built condition and a lower density of dislocations inside sub-grains. Thus, hardness and yield strength are reduced. Hardness of 316L SLM is of the same order of magnitude as forged 316LN and is explained by the high dislocations density even after annealing. A high content of nitrogen in the initial powder is not required. Compacting effect of the forward and backward counter-rotating roller could help increasing tensile properties.

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References

- [1] I.Tolosa et al. – « Study of mechanical properties of AISI 316 SS processed by SLM », following different manufacturing strategies » - Int J Adv Manuf Technol (2010) 51:639–647
- [2] K.Saeidi, Stainless steels fabricated by laser melting – Doctoral Thesis, Stockholm university, 2016
- [3] K.Akino et al. – « Strengths and Microstructure of SUS316L Fabricated by Selective Laser Melting » - J. Japan Inst. Met. Mater. Vol. 80, No. 12 (2016), pp. 772-777
- [4] M.L.Montero-Sistiaga et al., « Effect of heat treatment of 316L SS produced by SLM » - Proceed. 27th Annual Inter. Solid Freeform Fabrication Symp.- An AM Conference p.558-565
- [5] T.Niendorf et al. – « Process-microstructure-property relationships in Ni- and Fe-based alloys processed by AM » - Alloys for Additive Manufacturing Workshop, MPIE Düsseldorf, 4-5 July 2016
- [6] J.Kunz et al. – « Influence of HIP Post-Treatment on the Fatigue Strength of 316L-Steel Produced by SLM » - World PM2016 proceedings, Hamburg, Germany, 9-13 October 2016
- [7] NF EN ISO 6892-1 standard – « Metallic materials – Tensile testing – Part 1: Method of test at room temperature » - 12 November 2016
- [8] NF EN ISO 148-1 standard – « Metallic materials – Charpy pendulum impact test – Part 1: Test method » - 15 March 2017
- [9] NF EN 10028-7 Standard – “Flat products made of steels for pressure purposes – Part 7: Stainless steels” – 30 December 2016
- [10] ASTM A 240/A 240M – 04a standard - « Standard Specification for Cr and Cr-Ni Stainless Steel Plate, Sheet, and Strip for Pressure Vessels and for General Applications » - March 2004
- [11] B. Brown - « Characterization of 304L stainless steel by means of minimum input energy on the selective laser melting platform » - (2014), Masters Theses of Missouri University - Paper 7322