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Characterization of Additively Manufactured Parts of Alloy UNS N07718 in the API 6ACRA Chemical Composition

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ABSTRACT

After an era of prototyping, the additive manufacturing (AM) processes are currently passing through an industrialization period, when the production of real parts and complex field-applicable components is beginning to take part in the industry. Together with this fast development, the need to compare and evaluate the properties of additive manufactured products arises.

Among the most used wrought and additively manufactured nickel alloys in the oil & gas industry is the Alloy UNS(1) N07718, a precipitation hardening nickel-chromium-iron alloy enriched with niobium, molybdenum, titanium and aluminum, which confer to the material excellent mechanical properties combined with satisfactory corrosion resistance. Despite the significant application of this material, limited information is available on the literature regarding the properties achieved by the material on heat-treated printed parts.

In this context, the aim of these studies is to develop an optimized heat treatment for printed parts of Alloy UNS N07718 with chemical composition in accordance to the API(2) 6ACRA[1], printed in a SLM(3) machine and to characterize the properties obtained by the manufacturing process to get them compared to available data in the literature and to standard forged bars.

(1) Unified Numbering System for Metals and Alloys (UNS), SAE International, Warrendale, PA
(2) American Petroleum Institute (API), 1220 L St., N.W., Washington, D.C. 20005-4070
(3) Selective Laser Melting (SLM), Lübeck, Germany

Key words: UNS N07718, Additive Manufacturing, SLM, 3D printing, Mechanical Characterization

INTRODUCTION

The Selective Laser Melting (SLM) process is the most widely used process to fabricate additive manufactured parts of metallic materials. The SLM process enables the production of complex and customized parts for the different sectors of the industry, such as the aerospace, automotive, medical, and energy. By the application of this additive manufacturing method, a fully dense solid structure, reported in the literature to be greater than 99.7%,[2,3] is created through the selective melting of the surface of a metal powder bed by a laser beam, which grants structures with mechanical properties comparable to those achieved by wrought material when proper powder composition and printing parameters are selected.[2,4]

Besides the well known advantages of the use of additive manufacturing, like the possibility of producing small batches, more customized products, material savings and the easily reuse of waste material not used during the manufacturing, some authors also list the current challenges of this manufacturing process. Cost and speed of production, changing of designing, validation of mechanical and thermal properties, development of standardization and post processing, for example, are among the most challenging characteristics of AM implementation in the industry.[5]

Nickel alloys are important high technology metallic materials, since they can present good corrosion properties combined with outstanding mechanical resistance at both low and high temperatures. Among the most used wrought and additively manufactured nickel alloys in the oil & gas industry is the Alloy UNS N07718, a precipitation hardening nickel-chromium-iron alloy enriched with niobium, molybdenum, titanium and aluminum. Despite the significant application of this material, limited information is available on the literature regarding the properties achieved on AM post-manufactured parts with emphasis on improved microstructure and reduced anisotropy.

Wang Z. et all reported fine dendritic cast structures formed after additive manufacturing of Alloy UNS N07718 by SLM process and attributed the fine elongated structure to the high heat exchanges due to high laser energy densities.[3] Due to the energy transfer effect caused by the heat dissipation in the direction of the substrate, regular solidification structure on the building (vertical) negative z-direction is reported, composed by dendrites that grown parallel to the z-building direction. Similar structures were observed by X. Wang et all[4] and other authors. On the other hand, the cross section planes to the building direction often shows periodic morphologies resulting from the laser beam overlapping, which are characterized by larger equiaxed grains and dendritic arm spacing compared to single-pass regions.[2]

Usually, the dendritic structure is reported to be dissipated after carrying out the defined post-manufacturing heat treatment cycles and the area of equiaxed recrystallized grains drastically increases due to nucleation and grain growth.[2,3,4]

Some authors report mechanical properties at room temperature of additive manufactured parts of Alloy UNS N07718 comparable to those of solution annealed and age hardened wrought material.[3,4,6,7,8]

The present work evaluates the development of an optimized heat treatment for printed parts of Alloy UNS N07718 and the characterization of the properties obtained by the manufacturing and post-manufacturing process. The discussion of such information is of relevant importance to the further development and optimization of AM and post-AM processes and its future industrial application.

EXPERIMENTAL PROCEDURE

Material

The Alloy UNS N07718 powder material used in these studies was a mix of 4 VIM/AR (Vacuum Induction Melting / Argon atomization) heats with main chemical composition according to the API 6ACRA as shown on **Table 1**.

Table 1: Main chemical composition of powder heats

Heat	C	Cr	Ni	Mo	Ti	Nb	Fe	Al	B
P1	0.013	18.4	53.85	3.05	0.94	5.01	Balance	0.55	0.004
P2	0.011	18.4	54.24	3.07	0.95	5.06	Balance	0.5	0.004
P3	0.011	18.1	54.12	3.03	0.95	4.96	Balance	0.43	0.002
P4	0.011	18.3	53.77	3.02	0.95	5.02	Balance	0.46	0.003
API 6 ACRA for UNS N07718	Max 0.045	17.0-21.0	50-0-55-0	2.80-3.30	0.80-1.15	4.87-5.20	Balance	0.40-0.60	Max 0.0060

In order to define the printing parameters, 15 cubes were printed with different combinations of printing speed and power as exemplified on **Figure 1**.

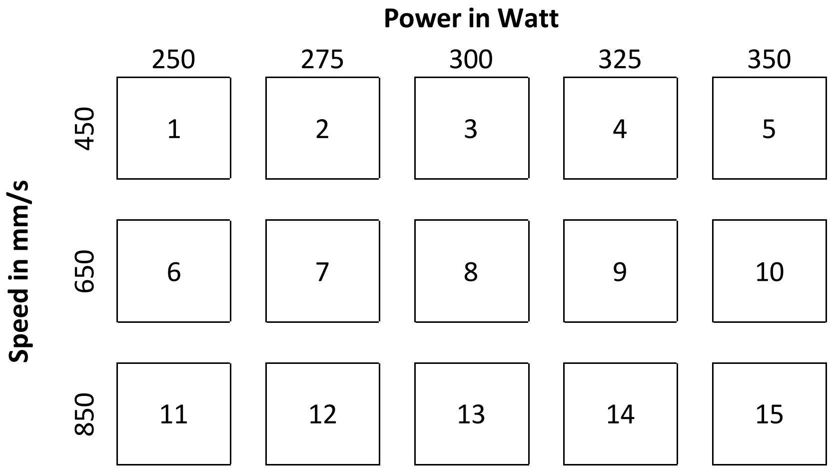

Figure 1: Scheme of printed cubes for parameter evaluation

After printing, each cube was analyzed for the presence of pores and/or lack-of-fusion defects. The combination of speed and power numbered 13 (850 mm/s, 300 W) with hatch 0.1 mm and layer thickness of 0.04 mm was chosen for printing the testing parts. The parts were printed using alternate 45°, 135° print strategy, as exemplified by **Figure 2**.

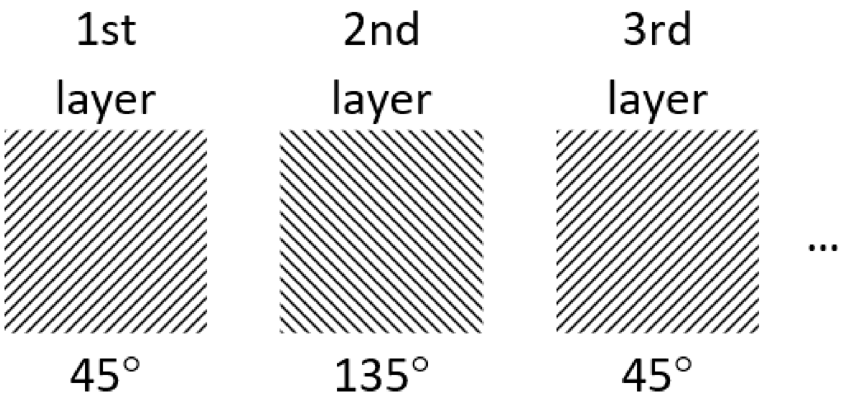

Figure 2: Schema of print strategy using alternate 45°, 135°

After definition of the printing parameters and strategy, raw samples were printed for the definition of heat treatment temperatures and testing program. The printed samples include parallelepiped samples, which were machined to testing samples with the desired dimensions.

The metallographic aspect of as-printed samples is shown on **Figure 3**. The dendritic cast structure and periodic morphology fit well with the structures described in the literature. Z. Wang et al.,[3] X. Wang et al.[4] and F. Cappuccini et al.,[7] reported to have achieved similar structures on as-printed samples of Alloy UNS N 07718. The porosity level is low with a slightly increase in regions close to the border of the samples.

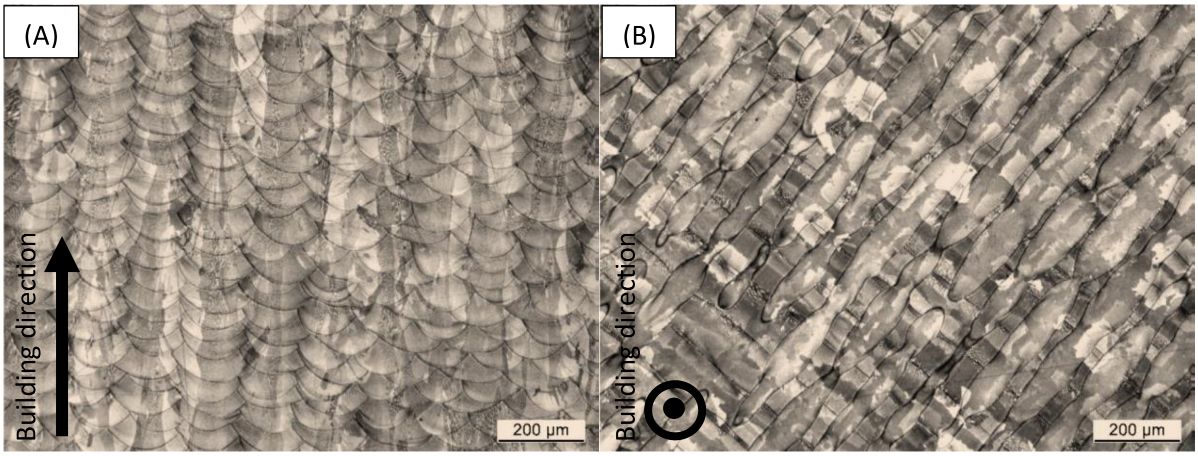

Figure 3: Optical images of as-printed samples etched with Kalling's No. 2

As the AMed parts do not pass through a forming process, it was expected that the energy required for grain recrystallization is higher in comparison to standard wrought material. In order to define the optimized solution annealing temperature, samples were solution heat treated at different temperatures: 1050 °C (1922 °F), 1100 °C (2012 °F) and 1150 °C (2102 °F) for 1 hour in a laboratory furnace (with air atmosphere) and cooled in water. After cooling, each sample had its microstructure checked for recrystallization and grain size. Both samples annealed at 1050 °C (1922 °F) and 1100 °C (2012 °F) were additionally age hardened and tensile tested in longitudinal and transversal directions to the building direction, in order to compare its mechanical properties to wrought Alloy UNS N07718 in the 120K Material Designation according to the API 6ACRA.

The obtained tensile properties are shown on **Figure 4**. Samples on longitudinal and transversal directions were tested in wrought material solution annealed at 1032 °C (1890 °F) and the corresponding "longitudinal" and "transversal" orientations to the building direction were tested in AMed material solution annealed at 1050 °C (1922 °F) and 1100 °C (2012 °F).

The temperature which lead to the best microstructure and closest tensile properties when compared to wrought Alloy UNS N07718 was selected (1050 °C, 1922 °F) and all of the remaining samples were solution annealed at this temperature followed by an age hardening heat treatment at 790 °C (1454 °F) for 7 hours – air cooling. This heat treatment sequence is capable to produce material according to the 120K Material Designation, as specified by the API 6ACRA.

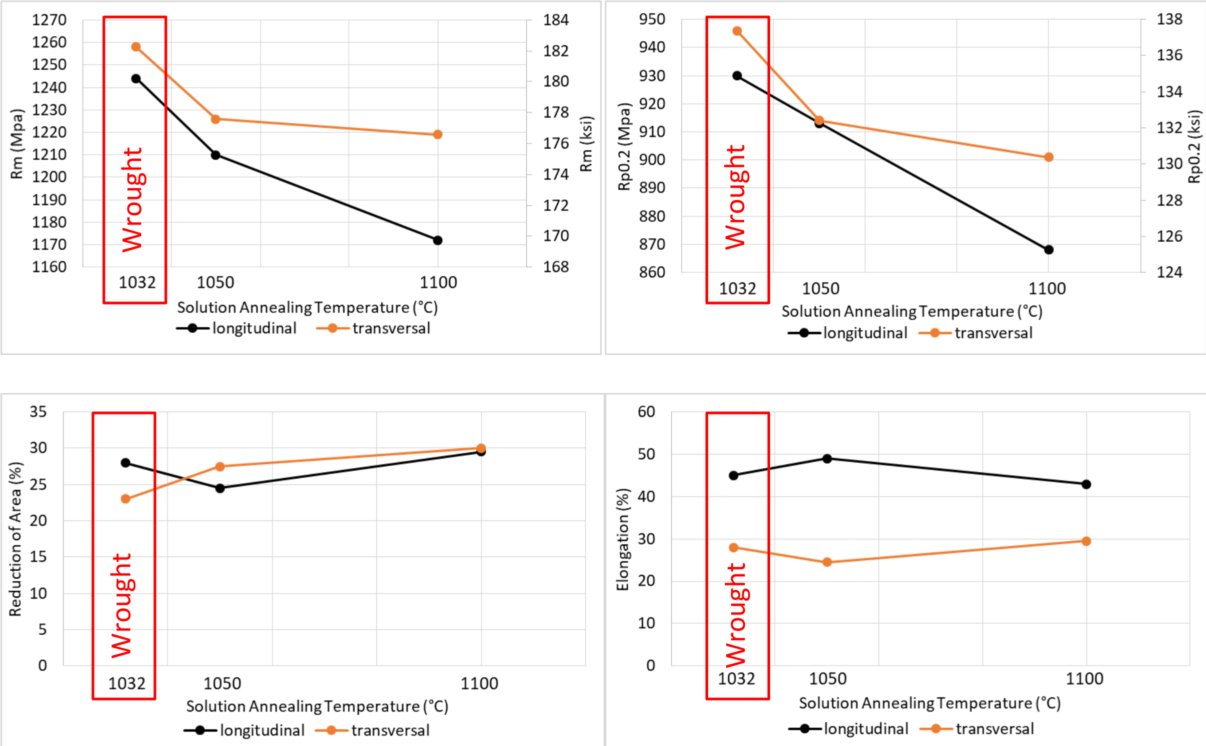

Figure 4: Tensile Properties of Wrought Alloy UNS N07718 (solution annealed at 1032°C, 1890 °F) in comparison with AMed parts solution annealed at 1050 °C (1922 °F) and 1100 °C (2012 °F) in longitudinal (black) and transversal (orange) directions. All samples were age hardened at 790 °C (1454 °F) after solution annealing to produce material according to API 6ACRA 120K Material Designation.

After final heat treatment, microstructural investigations were performed on mechanically polished and chemically etched specimens. Evaluation of the microstructure was performed using light optical microscopy techniques. The grain size was measured in accordance to ASTM(4) E112[9].

Mechanical Characterization

Tensile Testing

Tensile testing was conducted on smooth sub-size specimens B4x20 that were machined from the printed parallelepipeds. Samples were taken on the longitudinal and transversal to the building directions and tested according to the DIN EN ISO(5) 6892[10] at room temperature.

(4) American Society for Testing and Materials, West Conshohocken, Pennsylvania
(5) International Organization for Standardization, Geneva, Switzerland

Charpy Impact Testing

Charpy specimens with the notch in longitudinal and transverse directions to the building direction, corresponding to L-R and R-L orientations, were tested at a temperature of -60 °C (-76 °F) according to the DIN EN ISO 148[11]. Sub-size specimens were used to accommodate to the dimensions of printed parts and the impact energy was normalized per unit of area (σ), so that it could be directly compared to standard full size Charpy samples.

Hardness

Hardness test (HRC) was performed according to the DIN EN ISO 6508[12] in different positions on parallel and transversal plans to the building direction, according to the schema sketched on **Figure 5**.

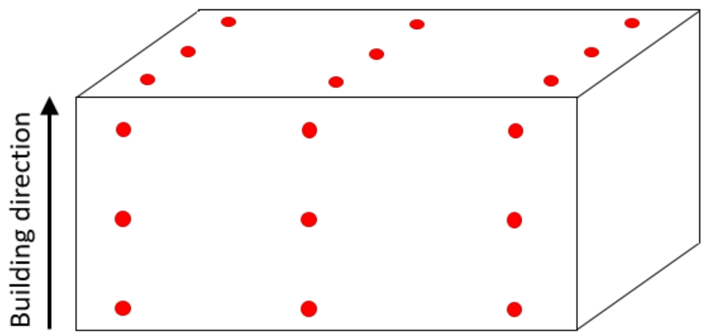

Figure 5: Schema of HRC hardness reading on AMed sample of Alloy UNS N07718

RESULTS

Microstructure

As explained above, the temperature of 1050 °C (1922 °F) was chosen as the optimal temperature to solution anneal the printed samples of Alloy UNS N07718, printed in a SLM machine using the printing parameters described above. The combination of a temperature of 1050 °C (1922 °F) and 1 hour of heat treatment was able to produce a full-recrystallized structure, with homogeneous grain size distribution. A hardening heat treatment given in the sequence at 790 °C (1454 °F) for 7 hours was capable to produce tensile properties comparable to wrought Alloy UNS N07718 bars. **Figure 6** shows an optical metallography of specimen polished and etched in oxalic acid. Several authors reported to have achieved the complete elimination of regular dendritic structure after heat treatment.[3,4,6,7,8]

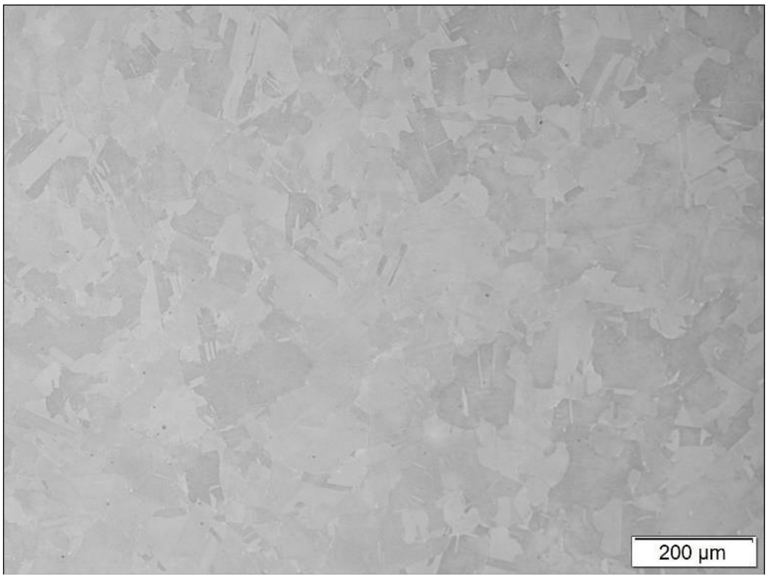

Figure 6: Optical metallography of printed specimen, solution annealed at 1050 °C and age hardened at 790 °C, polished and etched in oxalic acid.

After the chosen heat treatment cycle, the material is free from secondary phases and unacceptable grain-boundary precipitation and has an average grain size of ASTM number 3-3.5 (with average grain diameter 117 µm). Low porosity and melting failures caused by the Additive Manufacturing process could be identified and are not representative of the bulk microstructure.

Mechanical Characterization

Tensile Testing

The results of tensile testing at room temperature are shown on **Table 2** and go well with the API 6ACRA requirements for Alloy UNS N07718 120K Material Designation. The Yield Strengths are in average 889 MPa (129 ksi), what is usual for this material grade and there is a slightly tendency for lower yield and tensile strengths of samples transverse to the building orientation, when compared to samples taken parallel to the building direction.

The ductility is in agreement with the expected values and in accordance to the API 6ACRA for this material designation.

In the literature, the tensile properties of additive manufactured and heat treated samples of Alloy UNS N07718 present a huge variation. Cappuccini et al.[7] reported values of Yield Strength of AMed samples ranging from 637 to 799 MPa (92 to 116 ksi) and Ultimate Tensile Strength ranging from 1012 to 1101 MPa (147 to 160 ksi) depending on the sample direction. Ductility data was not published. On the other hand, Badrak et al.[14] achieved Yield Strengths of around 165 ksi and Tensile Strengths greater than 195 ksi. Elongation was between 14 and 17 % and Reduction of Area between 23 and 30 %.

Table 2: Tensile testing results of AMed samples of Alloy UNS N07718 at room temperature fabricated in the parallel and transversal directions to the building orientation

Sample orientation	Sample-Id.	Test temp.	Rp0.2		Rm		El.	RoA
		[°C]	[MPa]	[ksi]	[MPa]	[ksi]	[%]	[%]
Parallel to building direction	Z1H-1	23.1	904	131	1207	175	25.0	40
	Z2H-1	23.1	894	130	1224	178	26.5	44
Transversal to building direction	Z1V-1	23.1	877	127	1196	173	29.5	50
	Z2V-1	23.1	881	128	1194	173	29.5	47
API 6ACRA requirements for 120K Material Designation			**827-1000**	**120-145**	**Min 1034**	**Min 150**	**Min 20**	**Min 25**

<u>Charpy Impact Testing</u>

The results of Charpy impact testing are shown on **Table 3**. Sub-size 5x10 mm specimens were used to accommodate to the quantity of printed material and the impact energy was normalized per unit of area in the notch surface (σ). Note that, in terms of comparison, the impact energy required by the API 6 ACRA was also normalized as shown on **Table 3**.

Table 3: Charpy Impact results of AMed samples of Alloy UNS N07718 fabricated in the parallel and transversal directions to the building orientation

Sample orientation		Parallel to building direction - corresponding to L-R orientation	**API 6ACRA requirements**	Transversal to building direction - corresponding to R-L orientation	**API 6ACRA requirements**
Sample height [mm]		10	10	10	10
Sample thickness [mm]		5	10	5	10
Notch depth [mm]		2	2	2	2
Test temp. [°C]		-60	-60	-60	-60
Impact Energy [J]	M1	36	61	40	41
	M2	37		40	
	M3	40		37	
	Average	38	68	39	47
Normalized Impact Energy (σ)	M1	90	**76**	100	**51**
	M2	93		100	
	M3	100		93	
	Average	94	**85**	98	**59**

The normalized impact energies of Additive Manufactured samples are in accordance to the (normalized) requirements of the API 6ACRA and can be compared to values showed by wrought material in the 120K Material designation available in the literature.[13]

There is not much information available in the literature in regards to Charpy Impact toughness of AMed and heat treated Alloy UNS N07718, but it seems that several producers were not able to meet the requirements of API 6ACRA. [15]

Hardness

Several hardness indentations were made on printed samples on plans parallel and transversal to the building direction. **Table 4** summarizes all the hardness readings in HRC. The minimum value read is 34.9 HRC, the maximum value is 41.1 HRC and the readings lead to an average of 39.0 HRC. The results go well with the API 6ACRA requirements for Alloy UNS N07718 in the 120K Material Designation (32-40 HRC, with no individual hardness number greater than 2 HRC units above the maximum specified 40 HRC).

Authors reported in the literature hardness above 40 HRC for heat treated AM coupons.[14]

Table 4: HRC hardness readings on printed samples of Alloy UNS N07718 on plans parallel and transversal to the building direction

HRC		Side	Middle	Side	Average
transversal to building direction	Side	40.1	40.3	39.6	40.0
	Middle	39.8	40.2	38.4	39.5
	Side	35.2	36.1	34.9	35.4
parallel to building direction	up	39.1	40.2	39.4	39.6
	Middle	39	41.1	40.1	40.1
	bottom	39.5	39.4	39.5	39.5
	Average	38.8	39.6	38.7	**39.0**

A corrosion characterization of AM parts of Alloy UNS N07718 is ongoing and results will be discussed at the Corrosion conference 2021.

CONCLUSIONS

- Solution annealing of AMed parts of Alloy UNS N07718 require a higher annealing temperature than the standard wrought material to achieve 100% recrystallization and homogeneous microstructure and grain sizes.
- After correct solution annealing and age hardening, printed parts of alloy UNS N07718 are able to reach mechanical properties comparable to wrought material.

- Corrosion behavior of Alloy UNS N07718 is under investigation.

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