

Whitepaper

Property profile of stainless steels in additive manufacturing, produced on the TruPrint 2000

Content

Motivation and goal setting	4
Influence of elements in austenitic stainless steels	5
TruPrint 2000: Processing and test scope	6
Microstructural properties in comparison	7
Mechanical properties in comparison	8
Corrosion properties comparison	8
Conclusion and areas of application	0

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Abstract

In this whitepaper, two of the most important stainless steels 316L / 1.4404 and AlSi 630 / 17-4 PH / 1.4542 are to be compared in their most important properties. The comparison is complemented by a new Additive Manufacturing (AM) development, Printdur HSA, from Deutsche Edelstahlwerke Specialty Steel GmbH & Co. KG. As an introduction, an overview of the alloys available on the market is given and the main alloy systems are explained in more detail. The main focus of the material comparisons is the structure, the mechanical properties, and the corrosion properties.

Motivation and goal setting



Figure 1: Overview of the available stainless steels on the AM market

Stainless steels are a very large representative of steels on the world market. Alloys of their class can be found in every household in the form of cutlery. Likewise, stainless steels are used in industry in a wide variety of areas. Accordingly, this material class was focused in the development of additive manufacturing in order to achieve added value with the combination of the material and the new technology. The most prevalent representative of stainless steels in additive manufacturing is the material 1.4404, better known as 316L. The material belongs to the austenitic stainless steels. Due to the austenitic matrix, this material is particularly low in distortion and non-magnetic. The combination of these properties mean that 316L is very easy to process in additive manufacturing. Furthermore, the material has very good corrosion properties and good mechanical properties. Another representative of stainless steels is the material 1.4542, usually also referred to as 17-4 PH or AISI 630 (referred to as AISI 630 in the following). This is a ferritic-martensitic precipitation hardening steel with good corrosion properties for maritime applications. However, in order to achieve the desired properties, this material, unlike 316L, requires a heat treatment, which can be decided according to the corresponding ASTM A564/A564M standard. Heat treatments allows the user to achieve high strength with low ductility with the H900 variant. In contrast, the high temperatures of the H1150 heat treatment can optimize the properties in the low temperature application range.

Not only well-known stainless steels such as 316L and AISI 630 are gaining ground in the field of additive manufacturing, but also new developments are reaching market maturity and competing with well-known allovs. One of these variants is the Printdur HSA material from Deutsche Edelstahlwerke Specialty Steel GmbH & Co. KG. It is a fully austenitic stainless steel that achieves its properties without the use of carcinogenic elements, such as nickel and cobalt, and does not require subsequent heat treatments. It is also easy to work with and stands out for its excellent corrosion properties. An overview of the most common stainless steel alloys in the market is shown in Figure 1. The figure shows the comparison of alloys in the area of mechanical properties. In most cases, these are advertised with additions of hardness in the technical data sheets.

This is important because the mechanical properties are relevant for the design of the components, but more important key data for the target industries, such as the corrosion properties, are usually not included. For this reason, more extensive investigations of the 316L, AISI 630 and Printdur HSA alloys are carried out in their most common service conditions.

These include the metallographic properties (rel. density, microstructure and XRD results), the mechanical properties (tensile tests, notched bar impact tests and hardness) as well as various corrosion properties (surface corrosion according to ASTM G5, pitting corrosion according to ASTM G48 E). The aim is to provide the AM market with a

deeper understanding of the stainless steel variants and thus a better basis for making decisions on the choice of alloys. In addition, the fields of application are to be described and a classification of the alloys in different industries is to be made.

Alloy Element [WT-%]	Fe	с	Cr	Ni	Mn	Si	Мо	Cu	Nb+ Ta	N	Ρ	S
316L 1.4404	Rest	≤ 0.03	16.5 - 18.5	10.0 - 13.0	≤ 2.00	≤ 1.0	2.0 _ 2.5	-	-	≤ 0.11	< 0.045	< 0.015
AISI 630 1.4542 17-4 PH	Rest	≤ 0.07	15.0 _ 17.0	3.0 - 5.0	≤ 1.50	≤ 0.7	≤ 0.6	3.0 _ 5.0	≤ 0.45	-	≤ 0.04	< 0.015
Printdur HSA	Rest	0.4	18.0	< 0.1	21.0	≤ 0.2	2.0	-	-	0.6	≤ 0.02	≤ 0.02

Figure 2: Chemical compositions of the alloys used

Influence of elements in austenitic stainless steels

Based on the widely used material 316L, the typical alloy system of stainless steels will be explained below. The composition of the alloy can be divided into two categories. First, the corrosion resistance of the material must be ensured. This is achieved in particular by the element chromium. Like a chromium content (Cr) of > 12 wt.%, causes a dense chromium oxide layer to form on the material, which prevents corrosion reactions. The chromium oxide layer is further stabilized by the element molybdenum (Mo). In particular, the element Mo leads to increased resistance to pitting corrosion. Furthermore, in 316L the elements carbon and nitrogen are limited to a very low proportion (< 0.03 wt.%). This also serves to increase corrosion resistance, as both elements combined with the chromium present to create carbides. In this case, the bound chromium no longer helps to form the chromium oxide layer, which lowers the corrosion resistance. The addition of the element nickel leads to the formation of the non-magnetic austenitic microstructure in 316L. This means that the material can be used in many areas, such as medical technology. In order to achieve a non-magnetic microstructure, typical stainless steels require a nickel content of approximately 10%.

The influence of nickel on the magnetic behavior of steels is also evident when considering the chemical composition of magnetic AISI 630. The material has a nickel content of about 3-4%, which means that a non-magnetic microstructure is not achieved. Likewise, only chromium is used in AISI 630 to achieve corrosion resistance. The element molybdenum to increase resistance to pitting corrosion is omitted. As with 316L, the carbon content is severely limited (< 0.07 wt.%) to prevent

chromium from creating carbide. For this reason, niobium is also alloyed in AISI 630. Niobium has a higher affinity to carbon than chromium and thus binds off excess carbon.

Compared to 316L, AISI 630 exhibits very high mechanical properties. These high mechanical properties are adjusted in the downstream heat treatment. During heat treatment, the element copper lead to precipitation reactions that result in a hardening of the microstructure.

While 316L and AISI 630 are standardized steel grades which were developed for production as bars or castings and subsequently successfully tested for additive manufacturing, Printdur HSA developed specifically additive was for manufacturing. The Printdur HSA uses the addition of carbon and nitrogen to significantly increase the mechanical properties. Due to the rapid heating and cooling rates in 3D printing, precipitation of chromium-rich carbides or nitrides is avoided. Therefore, a reduction in corrosion resistance is avoided. The Printdur HSA also uses the elements chromium and molybdenum to achieve good corrosion resistance. In addition, nitrogen, in the present dissolved state, increases corrosion resistance to pitting corrosion.

Like 316L, Printdur HSA has a non-magnetic austenitic structure. This is not achieved by the element nickel, but by the combination of the element's manganese, carbon and nitrogen. This results in a nickel-free stainless austenitic steel.

TruPrint 2000: Processing and test scope

The TruPrint 2000 celebrated its market debut at Formnext 2020 and has since become a reliable and important machine in the TRUMPF Additive Manufacturing portfolio. The machine closes the gap between the small-format TruPrint 1000, which shows its strengths in the dental and jewelry industries, and the TruPrint 3000 and 5000 medium-format machines at the upper end, which were designed for large components in series production. The TruPrint 2000 benefits as a link, with a build envelope of 200 mm in diameter and a maximum build envelope height of 200 mm. The small focus diameter of 55 µm, which is equipped as a full-field multilaser, enables the highest quality standards for surface properties and reliable productivity. In addition, the system is equipped with extensive process monitoring options, such as condition, powder bed and melt pool monitoring, to leave no doubt about quality assurance. The inert, closed powder circuit also supports the quality demands of the components. This means that the powder remains in an inert gas atmosphere throughout the entire process, which in turn enables the powders to be recycled, even with critical materials.

Basic parameters of the three different materials 316L, AISI 630 and Printdur HSA are developed on the TruPrint 2000 which the following testing was completed. A 40 μ m powder layer thickness was used as the basis for parameter development. Subsequently, a build job layout is defined which represents all analysis geometries. In order to maintain the comparability of the alloys, the same build job was carried out with each material. The setup can be seen in Figure 4.

In the layout, care was taken to ensure that all test geometries were distributed over the build plate so that statements about the homogeneity of the build plate can be made.

TruPrint 2000					
Built volume (cylinder)	mm x mm	Ø 200 x H 200			
Processable materials		Weldable metals in powder form, such as: Stainless steels, tool steels, aluminium, nickel-based, cobalt- chrome, titanium alloys, or amorphous metals			
Layer thickness	μm	20 - 100			
Max. laser powder at the workpiece (TRUMPF fiber laser)	w	300 Optional multilaser: 2 x 300			
Beam diameter	μm	55			
Scan speed (powder bed)	m/s	Max. 3			
Preheating	°C	Up to 200			
Unpacking in the machine		Integrated powder conveyor			
Shielding gas		Nitrogen, argon			
Power supply	V / A / Hz	400/460 - 32 - 50/60			
Dimensions	mm	2180 x 2030 x 1400			
Weight	kg	3200			



Figure 3: Specification of the TruPrint 2000

Figure 4: Overview of the construction job layout used

Microstructural properties in comparison

For the characterization of the relative density, transverse sections of the specimens were prepared, which were subsequently evaluated with an optical microscope and analysis software. All three materials, in combination with their developed parameter, achieved a relative density greater than 99.9% over the complete build plate.

The transverse sections of the alloys were then repolished and subjected to Adler dip etching to reveal the microstructural constituents.

The most commonly used material, 316L stainless steel, has rod crystalline grains that grow along the direction of construction and thus follow the thermal gradient of solidification. The composition of the alloy gives it an austenitic solidification form, which can be looked up by the Schäffler diagram or recalculated by the underlying Cr and Ni equivalents. This is also found in the microstructure and can also be confirmed in the additionally performed XRD measurements, which show a 95% austenitic microstructure. The remaining 5% do not show a clear assignable peak, but according to the literature, the compositiondependent and the imbalances solidification sequence can be assigned to the delta ferrite.

The material AISI 630 shows а different microstructure compared to 316L. The microstructure formed after the process was refined in the heat treatment process, so that martensiticferritic structures have formed in the primary solidified y-grains. The XRD measurements confirm a 97% emerging ferritic-martensitic microstructure, with a residual austenite of 3% detected.

In the case of the Printdur HSA material, the melt beads typical of the LPBF process are clearly visible in the etched state. Austenitic grains or grain boundaries cannot be determined. The X-Ray diffraction (XRD) measurements carried out show a 98% austenitic microstructure. Despite the high carbon and nitrogen content, no chromium-rich carbides or nitrides can be detected. This confirms that the fast-cooling rate in the LPBF process results in a very fine, precipitation-free austenitic microstructure in the Printdur HSA.







Figure 5-7: Light microscope images of the alloys used. All images were taken with a magnification of 100x.

Mechanical properties in comparison

For the investigation of the mechanical properties, Vickers measurements according to DIN EN ISO 6507, tensile tests in standing form according to DIN EN ISO 6892-1:2017 with a specimen geometry according to DIN 50125:2016 (Form A) and notched bar impact tests according to DIN EN ISO 148-1 :2017-05 were performed. All results of the tests can be seen in Figure 8.

The hardness measurements according to Vickers confirm the findings from the metallographic investigations. 316L has a very low hardness of 242 \pm 3 HV, which is due to the alloy composition. The yield strength and tensile strength are also lowest for 316L compared to the other alloys investigated. The yield strength is 504 \pm 10 MPa and the tensile strength is 640 \pm 4 MPa. An advantage of the 316L alloy is its high elongation at fracture, which has the highest value of 40 \pm 1% in comparison. The high elongation at fracture of the

alloy is also reflected in the impact strength. Here, 316L is at the top with 148 \pm 7 J.

In comparison, the alloy AISI 630 exhibits the highest hardness 445 ± 3 HV and strength 1419 ± 14 MPa. There are two reasons for this, martensitic structure (See Fig. 6) and the precipitated hardening in heat treatment. In contrast, the elongation at fracture and the fracture toughness are obtained, which in this alloy is very low compared to the others at 9 ± 2 %.

A good combination of high strength/hardness and ductility is offered by the alloy Printdur HSA. The hardness values are 374 ± 5 HV and the tensile strength is 1116 ± 6 MPa, which is only about 300 MPa lower than the strength of AISI 630. The reasons for this are the enrichment of the carbon and nitrogen content in the alloy, which in turn also leads to very good ductility of the material.

Material		316L	AISI 630	Printdur HSA	
Heat Treatment		None	H900	None	
Hardness according to Vickers [HV]		242 ± 3	445 ± 3	374 ± 5	
	Rp _{0,2} [MPa]	504 ± 10	1147 ± 55	861 ± 13	
Tensile Testing	R _m [MPa]	640 ± 4	1419 ± 14	1116 ± 6	
loomig	A [%]	40 ± 1	9 ± 2	30 ± 5	
Fracture toughness [J]		148 ± 7	9 ± 1	23 ± 4	

Figure 8: Mechanical properties of the alloys used.

Corrosion properties comparison

The most important property of stainless steels is their corrosion resistance. To determine this, the resistance to surface corrosion was first measured according to ASTM G5 in 0.5 molar sulfuric acid. All three stainless steels investigated exhibited identical positive to surface corrosion.

Different corrosion behaviors of the steels were evident with respect to resistance to pitting corrosion, which was determined according to ASTM G48, Method A. In this test, the specimens are weighed before the measurement begins. Subsequently, the specimens are exposed to a ferric chloride bath at different temperatures 5°C,

15°C, 25°C, 30°C and 35°C for a defined time. Subsequently, the specimens are weighed again so that a removal rate/corrosion rate can be determined. The results of the tests can be seen in Figure 9 and Figure 10. Figure 10 is composed of the five temperatures investigated and the three materials, so that 316L is found in the upper third, AISI 630 in the middle third and Printdur HSA in the lower third. Clear differences can be seen between the three materials. In the temperature range of 5 °C, the corrosion rate is as follows among the materials: Printdur HSA < 316L < AISI 630. From the next higher temperature range of 15 °C, the behavior changes and the corrosion rate of 316L exceeds that of AISI 630. As the temperatures continue to rise, the corrosion rates are defined as follows. Printdur HSA < 630 < 316L. Printdur HSA shows a low corrosion rate over the complete temperature range, with a first steeper increase between 30-35 °C.

The different properties with regard to pitting corrosion correlate with the so-called Pitting Resistant Equivalent Number (PREN). This index evaluates the resistance of an alloy to pitting corrosion on the basis of the alloy composition. The PREN is defined as follows:

PREN= %Cr + 3.3 x %Mo + 16 x %N

The higher the PREN value, the more resistant the alloy is to pitting corrosion.

PREN also explains why Printdur HSA has

significantly better properties than ALSI 630 and 316L: The higher corrosion resistance against pitting corrosion of the Printdur HSA can be attributed to the high nitrogen content. The nitrogen dissolved in the metal matrix leads to reduced pitting, which increases the corrosion resistance. Overall, the results show that with regard to surface

corrosion, which occurs for example in acidic media, the three stainless steels investigated exhibit identical properties. However, in the area of pitting corrosion, which occurs e.g. in seawater or in typical dishwashers, significant differences can be determined.



Figure 9: Corrosion rate of the alloys at different test temperatures



Figure 10: Tested samples according to ASTM G48 (resistance to pitting corrosion). The figure is divided horizontally in three (316L, AISI 630, Printdur HSA) and vertically 5 (5°C, 15 °C, 25 °C, 30 °C 35 °C).

Conclusion and areas of application

Due to their easy processability in additive manufacturing, stainless steels are widely established in industry. In this whitepaper, it exibits that the properties of components can be optimized for the application by using different alloys. All the results obtained are again summarized in Figure 11. 316L scores with very good processability and also good corrosion resistance. However, the material has the lowest mechanical properties. The properties and processability of 316L make it a good application for a variet of industries, including food, chemical sanitary industries, and general mechanical enginering.

If higher mechanical properties are required, Printdur HSA can be used.

Printdur HSA also has the best properties in terms of pitting corrosion, and can be used in off-shore applications, the chemical industry, the oil and gas industry. Due to absence of the elements nickel and cobalt, this material offers advantages in the safety classification and handling of the powder The material can also be purchased manufactured in accordance with DIN:EN ISO 13485, which means that it can be used in medical technology.

AISI 630 scores with very high mechanical properties. To achieve these, however, heat treatment must be carried out. Furthermore, it must be taken into account that the AISI 630 is ferromagnetic in contrast to the 316L and Printdur HSA. The material is mainly used in medical technology, although many other industries, such as the chemical industry or the oil and gas industry, offer possible applications.

Material		316L	AISI 630	Printdur HSA	
Relative Density [%]		> 99.7 %	> 99.7 %	> 99.7 %	
Safety (REACH, UN)		Co free	Co free	Ni / Co free	
Heat Treatment		No	H900	No	
	Rp _{0,2} [MPa]	504 ± 10 MPa	1147 ± 55 MPa	861 ± 13 MPa	
Tensile Test	R _m [MPa]	640 ± 4 MPa	1419 ± 14 MPa	1116 ± 6 MPa	
	A [%]	40 ± 1 %	40 ± 1 % 9 ± 2 %		
Impact Test [J]		148 ± 7 J	9 ± 1 J	23 ± 4 J	
Application Temperature [°C]		Up to 550 °C	Up to 315 °C	Up to 200 – 300 °C	
XRD		95% Austenite*	97% Ferritic 3% Austenitie	98 % Austenite*	
Surface Corrosion ASTM G5 I_{δ} Potential Curve (H ₂ SO ₄)		✓	√	~	
Corrosion	Pitting Corrosion ASTM G 48 E (FeCl ₃ , 35 °C)	48.9 mm/a	27,6 mm/a	5.9 mm/a	
	Pitting Equivalent Number (PREN	24	16	34	

Figure 11: Summary of the results obtained * No further peaks are assignable

For more information on available materials, please visit: <u>Metal powder for additive manufacturing | TRUMPF</u>

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