

Development of 316L, 2507 Duplex and Nickel-Free Stainless Steel for the Binder Jet Process

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ABSTRACT

Nickel-containing stainless steels can be problematic in applications that include skin contact due to potential allergic reactions. There is currently a limited availability of materials optimized for the binder jet process, thus development of a material is needed that can be used with this process for applications where nickel allergies may be a concern. For this study, material properties of binder jet test specimens were investigated using three stainless steels containing varying nickel concentrations: 316L, 2507 duplex and nickel-free stainless steel. Mechanical properties are evaluated in the sintered and heat treated condition. Microstructures and porosity are discussed in relation to the sintering conditions, mechanical properties, and corrosion resistance.

INTRODUCTION

As the binder jet process continues to be evaluated for different part applications new materials need to be qualified for use with this system. This process works by depositing powder layer by layer and applying a liquid binding agent that holds the powder particles together. The binder jet parts are taken through debinding and sintering where densification occurs. Standard alloy grades need to be tailored for this additive manufacturing (AM) technique due to differences in the starting density from other powder metallurgy processes like metal injection molding (MIM). To further improve the binder jet part final density liquid phase sintering or hot isostatic pressing can also be used. High volume part manufacturing is possible with binder jetting due to the separation of the printing and sintering steps. This process also allows for a wider range of printable materials than compared with other AM processes such as laser powder bed fusion, which is limited to weldable materials.¹

Stainless steels have high strength, good corrosion and oxidation resistance.² They are used in a range of applications including heat exchangers, furnace parts and boilers.³ The different types of these steels include austenitic, duplex, ferritic, martensitic and precipitation hardening and they contain at least 11% chromium.⁴ To obtain a fully austenitic stainless steel high amounts of nickel, carbon, nitrogen and manganese are needed to make austenite stable at room temperature. Alternatively, to obtain a ferritic

stainless steel there must be enough ferrite stabilizers such as chromium and silicon to make the crystal structure BCC at room temperature.⁴

A commonly used austenitic stainless steel is AISI 316L. It has at least 16% chromium and 10% nickel, providing it with good corrosion resistance. For jewelry or wearable applications using an alloy with a high level of nickel such as 316L may cause skin irritation due to the potential of corrosion of the metal and release of metal ions when in contact with body fluids.^{4,5} Thus, there is a need to qualify a stainless steel with a low concentration of nickel and good corrosion resistance for use in the binder jet process that will be suited for these types of applications. Nickel-free stainless steel is also austenitic, non-magnetic and has good corrosion resistance. In this steel nitrogen is used instead of nickel to make the austenite stable at room temperature, while providing improved strength.² In its wrought form, this steel can be used for applications such as surgical implants.⁶

2507 duplex is another stainless steel that has a good mechanical properties and corrosion resistance and has a lower concentration of nickel than 316L. Its microstructure contains a mixture of ferrite and austenite which provides a good balance of properties.⁴ Duplex stainless steels are commonly used in different industries including oil and gas and are available in various forms including bar forgings, castings, and sheet metal.³ Secondary phases such as chromium nitrides and sigma phase are more likely to precipitate in duplex steels due to their higher molybdenum and chromium contents.⁷ When these phases precipitate the adjacent regions become depleted in chromium making localized corrosion likely to occur. The steel can be heated to a high temperature to dissolve these phases and rapidly cooled.³

When sintering conventional PM stainless steels with hydrogen, oxides in and on the powder will reduce, which results in better ductility, yield strength, tensile strength and density.² Sintering in nitrogen will cause nitriding to take place due to chromium's affinity for nitrogen causing increased yield strength, tensile strength, hardness but lower elongation. This may also reduce the overall corrosion resistance because chromium will be depleted in regions.² The sintering cycle used for 316L must be adapted to obtain a high density as well as a high concentration of nitrogen as required by 2507 duplex and nickel-free stainless steel to achieve the desired microstructure.

EXPERIMENTAL PROCEDURE

Powder Characterization

The powders used in this study were gas atomized with nitrogen and screened to <25 micron particle size distributions. Table I shows the chemical compositions of the powders as well as the equivalent wrought alloy specification. The 2507 duplex powder has almost half of the nickel content of the 316L powder and it also has the highest amount of chromium. This higher chromium content helps to stabilize ferrite while its higher nitrogen content acts to stabilize austenite. The nickel-free stainless steel has trace amounts of nickel and increased manganese content as compared to the 316L powder. The nitrogen content of the nickel-free stainless steel is the highest of the three alloys which helps to stabilize austenite.

Table I. Chemical compositions of the powders used in this study and the UNS wrought equivalents (wt.%).

Material	Ni	Cr	Mn	Mo	Si	N	C	O
UNS S31603	10-14	16-18	<2.0	2-3	<0.75	<0.1	<0.03	---
316L Powder	11.50	17.86	1.42	2.57	0.48	0.06	0.01	0.09
UNS S32750	6-8	24-26	<1.2	3-5	<0.8	0.24-0.32	<0.03	---
2507 Powder	6.50	25.40	1.07	3.80	0.53	0.29	0.02	0.02
UNS S29225	<0.05	16.50-18	9.5-12.5	2.7-3.7	0.2-0.6	0.45-0.55	0.15-0.25	---
Ni-Free SS Powder	0.17	17.35	10.85	3.47	0.83	0.40	0.02	0.11

* Each alloy has a balance of iron

Scanning electron microscopy (SEM), using a JEOL 6460 LV SEM, was used to evaluate the powder morphology. Carbon, oxygen and nitrogen content were measured by LECO analysis. Apparent density and tap density were measured following MPIF standards 28 and 46.⁸ Particle size was measured using a Sympatec Helos BF laser particle-size analyzer in accordance with ASTM B822.

Binder Jet Processing

Mechanical testing samples (MPIF Standard 10 conventional flat dogbone), transverse rupture strength (MPIF Standard 41) samples for density evaluation and green strength (MPIF Standard 15) samples were printed with a 50 μ m layer thickness in the XY plane on a HP Multi Jet Fusion Printer with a water-based binder. As-built samples were measured for green density and green strength.

Sintering and Heat Treatment

Samples were sintered at DSH Technologies utilizing MIM3045T furnaces from Elnik Systems. With this system the thermal debind and sinter process is combined in one furnace. The equipment has a maximum temperature of 1600°C with partial pressure or vacuum control. It has an all-metal process zone with atmosphere capabilities of pure hydrogen, nitrogen, argon, or vacuum environments. Different sintering cycles were used where the temperature, time at temperature and atmosphere were varied. A batch furnace with a maximum temperature of 1150°C and nitrogen atmosphere was used to heat treat the samples. Tensile testing following MPIF Standard 10 and apparent hardness (MPIF Standard 43) measurements were made on sintered and heat treated samples.

Metallographic Analysis

Standard metallographic techniques were used to evaluate the microstructure of the as-sintered and heat treated samples. Light optical metallographic (LOM) images were taken of the etched sintered and heat treated sample cross-sections perpendicular to the build direction. Local chemical composition analysis was completed using a JEOL 6460 LV SEM with a Thermo Fisher Scientific Energy Dispersive Spectrometer (EDS). Pathfinder software was used to analyze the chemical analysis data. Conditions for the EDS analysis were: accelerating voltage of 15 keV, 2kx magnification, live acquisition time of 60 seconds at 30% dead time, spot analysis with at least 5 spots was analyzed for the microstructural constituents.

RESULTS & DISCUSSION

Powder Characterization

Figure 1 shows an SEM micrograph of the nickel-free stainless steel powder. The particles are very spherical with few satellites. This powder morphology is similar for the 316L and 2507 stainless steel powders. This powder shape is beneficial to the binder jet process because it leads to better flow during spreading and improved packing in the bed. The shape of the particles affects various powder properties including apparent density, flow and compressibility.²

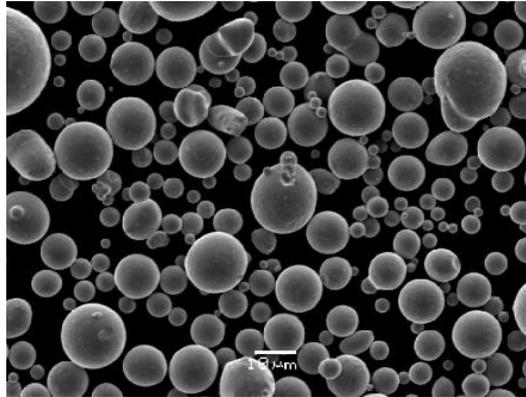


Figure 1. SEM micrograph of the nickel-free stainless steel powder

Table II shows measured properties of the powder including particle size, apparent density, and tap density. The powders showed similar particle size distributions and tap densities.

Table II. Powder properties

Material	d ₁₀ [μm]	d ₅₀ [μm]	d ₉₀ [μm]	Apparent Density [g/cm^3]	Tap Density [g/cm^3]
316L	5.6	14.1	24.1	4.09	4.72
2507	5.2	11.7	23.5	3.93	4.77
Ni-Free SS	4.9	11.7	21.8	4.11	4.79

Green Properties

Table III shows the as-built properties of the different alloys after printing. The green densities (GD) achieved were slightly higher than the apparent densities of the powders. Green strength (GS) was measured to determine the resistance of the material to breakage during handling.²

Table III. As-built properties of the binder jet samples

Material	GD [g/cm^3]	GS [MPa]
316L	4.66	6.0
2507	4.56	6.5
Ni-Free SS	4.19	7.9

Mechanical Properties – 316L Stainless Steel

316L binder jet samples were sintered at 1380°C for 2 hours under two different atmospheres to examine the effect of the atmosphere on the material properties. Table IV shows the mechanical properties of the as-sintered samples as well as wrought, MIM and press/sinter (PM) specifications for 316L. The samples sintered in the hydrogen atmosphere (Table IV-a) achieved a high density and exceeded the wrought and MIM 316L specifications for yield strength (YS), ultimate tensile strength (UTS) and elongation. The

samples sintered in the 95 vol.% N₂ / 5 vol.% H₂ atmosphere (Table IV-b) exceeded the PM specification for SS-316N2 (MPIF Standard 35-SP) for all the properties at a similar density. A higher UTS and YS was reached after sintering in the nitrogen atmosphere due to the effect of nitrogen alloying.⁹ A higher density and elongation was reached after sintering in the reducing hydrogen atmosphere.

Table IV. Mechanical properties of as-sintered 316L binder jet samples and specifications for wrought, MIM and PM 316L.

316L Material	Density [g/cm ³]	N [%]	0.2% YS [MPa]	UTS [MPa]	Elong. [%]	Apparent Hardness [HRA]
UNS S31603, Annealed	---	<0.1	170 min	485 min	40 min	58 max
MIM-316L, As-sintered	7.6	---	140 min	450 min	40 min	43
Binder jet, Sintered in hydrogen (a)	7.84	0.004	197	569	49	41
PM SS-316N2, As-sintered	6.5	0.2-0.6	230 min	410	5 min	41
Binder jet, Sintered in 95%N ₂ /5%H ₂ (b)	6.52	0.870	394	634	6	44

(a) Sintered at 1380 °C for 2hrs in hydrogen atmosphere

(b) Sintered at 1380 °C for 2hrs in 95 vol.% N₂ / 5 vol.% H₂ atmosphere

Figure 2a shows the etched microstructure of the 316L samples sintered in a hydrogen atmosphere at 1380°C for 2 hours. The primary phases are austenite and delta ferrite shown by the light areas and grey areas respectively. There is also a secondary phase along the ferrite/austenite grain boundaries. This secondary phase may form during slow cooling if there is carbon or nitrogen in the stainless steel, which are slower to diffuse in ferrite than austenite.² The presence of these precipitates can lead to intergranular corrosion so rapid cooling from the sintering temperature should be used to suppress the formation of these precipitates or alternatively a solution anneal can be used to dissolve them.^{2,4} Figure 2b shows the microstructure of a 316L sample that was heat treated at 1093°C for 30 minutes and then water quenched. The secondary phase seen at the grain boundaries in the as-sintered sample was dissolved. The grains are equiaxed and homogenous and there are twin boundaries present.

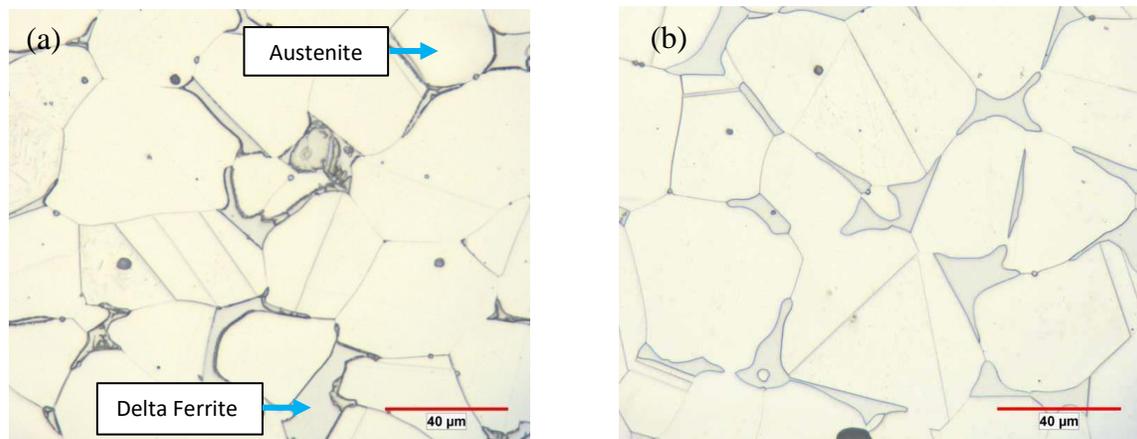


Figure 2. LOM image showing microstructure of (a) 316L as-sintered: 1380°C for 2hrs in a hydrogen atmosphere and (b) 316L sintered and heat treated: 1093°C for 30 minutes in a nitrogen atmosphere and water quenched. (etchant Glyceregia)

Figure 3 shows the etched microstructure of the 316L samples sintered in an atmosphere of 95 vol.% N₂ / 5 vol.% H₂ at 1380°C for 2 hours. The light areas are austenite and the dark areas with texture are pearlite formed due to the high level of nitrogen in the sample. The nitrogen picked up from the atmosphere provides strengthening, but without the reducing hydrogen atmosphere densification will be inhibited as seen by the increased porosity content. When sintering stainless steel in a nitrogen atmosphere it will result in an uptake of nitrogen causing nitride formation which will cause chromium depleted regions thereby reducing the overall corrosion resistance.

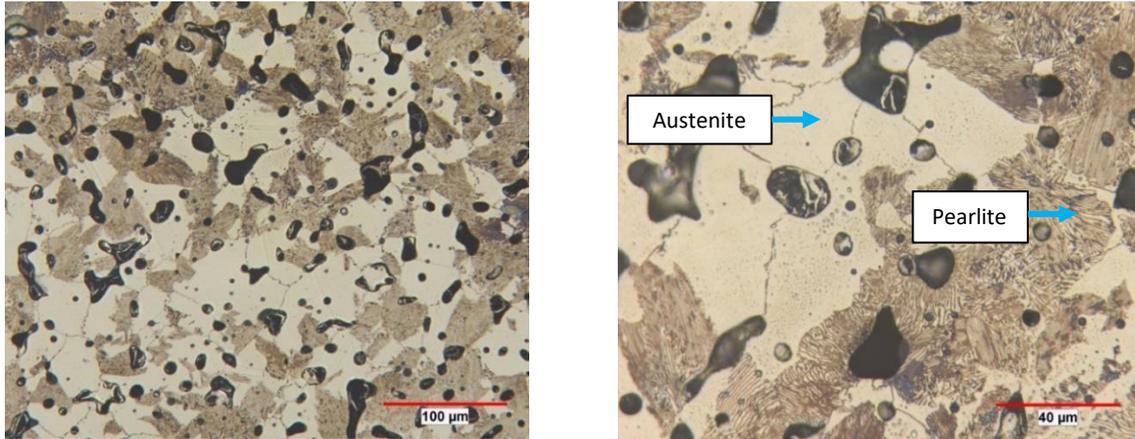


Figure 3. LOM images at two different magnifications showing the microstructure of 316L as-sintered: 1380°C for 2hrs in 95 vol.% N₂ / 5 vol.% H₂. (etchant Glyceregia)

Mechanical Properties – 2507 Duplex Stainless Steel

2507 duplex binder jet samples were sintered at two different temperatures and atmospheres to evaluate the effect on the material properties. Samples were sintered at 1380°C for 2 hours in a hydrogen atmosphere and then heat treated. For comparison samples were also sintered for 2 hours in an atmosphere of 95 vol.% N₂ / 5 vol.% H₂. Higher nitrogen content in this alloy can lower the liquidus temperature, so 1365°C was used as the sintering temperature in this cycle. Table V shows the mechanical properties of the as-sintered and heat treated samples.

The samples sintered in the hydrogen atmosphere (Table V-a) had very little nitrogen content and the elongation and UTS were lower than the wrought specification. A solution anneal was performed at 1149°C for 4 hours in a nitrogen atmosphere followed by water quenching. This heat treatment led to improved mechanical properties and the wrought minimum property specifications were met (Table V-b).

The 2507 duplex samples sintered in the 95 vol.% N₂ / 5 vol.% H₂ atmosphere achieved a nitrogen level of 0.27% and this increase in nitrogen caused a liquid phase to form and almost full density was reached (Table V-c). The sintered samples were heat treated into the austenite-ferrite phase region at 1093°C for 1 hour in a nitrogen atmosphere then water quenched to achieve a duplex microstructure. The heat treated samples (Table V-d) were tested for mechanical properties and showed improved YS, UTS, elongation, and hardness from the as-sintered condition. To compare the mechanical testing results between the three alloys by binder jet, 2507 duplex samples can be sintered in a nitrogen atmosphere at a temperature below 1365°C to avoid the formation of a liquid phase, but still achieve the targeted nitrogen content.

Table V. Mechanical properties of 2507 binder jet samples and the specification for the UNS wrought equivalent.

2507 Material	Density [g/cm ³]	N [%]	0.2%YS [MPa]	UTS [MPa]	Elong. [%]	Apparent Hardness [HRA]
UNS S32750, Annealed	---	0.24-0.32	550 min	795 min	15 min	67 max
Binder Jet, Sintered under hydrogen (a)	7.35	0.01	594	699	3	54
Binder Jet, Sintered and Heat Treated (b)		---	625	794	15	58
Binder Jet, Sintered under 95% N ₂ /5% H ₂ (c)	7.75	0.27	534	780	19	59
Binder Jet, Sintered and Heat Treated (d)		---	576	852	30	62

(a) Sintered at 1380°C for 2hrs in hydrogen atmosphere

(b) Sintered at 1380°C for 2hrs in hydrogen atmosphere and heat treated at 1149°C for 4hrs in nitrogen atmosphere then water quenched

(c) Sintered at 1365°C for 2hrs in 95 vol.% N₂/ 5 vol.% H₂ atmosphere

(d) Sintered at 1365°C for 2hrs in 95 vol.% N₂/ 5 vol.% H₂ atmosphere and heat treated at 1093°C for 1hr in nitrogen atmosphere then water quenched

Figure 4 shows the etched microstructure of the binder jet 2507 samples sintered in the 95 vol.% N₂ / 5 vol.% H₂ atmosphere. The light areas are austenite and the grey areas are ferrite. There is a secondary phase at the austenite/ferrite grain boundaries.¹⁰ Sigma phase is a nonmagnetic intermetallic phase that mostly precipitates at ferrite/ferrite and ferrite/austenite grain boundaries due to slow cooling. It is detrimental to mechanical properties and corrosion resistance, but a solution anneal can be used to dissolve this phase and improve these properties.

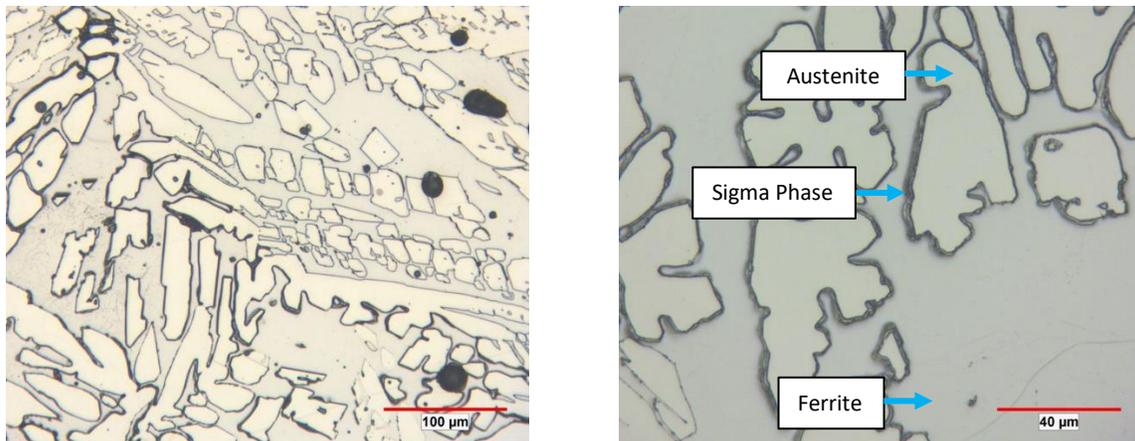


Figure 4. LOM images at two different magnifications showing the microstructure of 2507 duplex sintered at 1365°C for 2hrs in 95 vol.% N₂ / 5 vol.% H₂ atmosphere. (etchant Glyceregia)

Table VI shows the chemical composition of the phases present in the microstructure of the 2507 duplex samples sintered in the 95 vol.% N₂ / 5 vol.% H₂ atmosphere. The phases correspond to the areas highlighted in Figure 4. The austenite stabilizers (Ni and Mn) are increased in the austenite and the ferrite

stabilizers (Cr, Mo and Si) are increased in the ferrite. The sigma phase has the highest concentration of chromium and molybdenum leaving the adjacent regions to this phase depleted of these elements and deteriorating the corrosion resistance.

Table VI. Chemical composition of microstructural constituents (wt.%)

Phase	Cr	Ni	Mo	Mn	Si	Fe
Austenite	25.18	7.45	3.09	1.39	0.76	62.14
Ferrite	27.23	4.61	4.58	1.32	0.88	61.39
Sigma	30.02	4.28	7.90	1.22	1.07	55.51

Figure 5 shows the microstructure of the 2507 duplex samples that were sintered in the 95 vol.% N₂/ 5 vol.% H₂ atmosphere and then heat treated at 1093°C for 2 hours in a nitrogen atmosphere and water quenched. A duplex structure was achieved after solution annealing and no secondary phase was found at the grain boundaries or inside the ferrite grains. The ferrite is shown by the darker elongated pools within the lighter austenite matrix. Equal amounts of ferrite and austenite will achieve the best balance of properties in duplex stainless steels. To adjust the amount of these phases different heat treating temperatures can be used and longer times at temperature may be needed to reach equilibrium in the sample. These heat treated samples should have improved corrosion resistance as compared to the as-sintered samples due to the dissolution of sigma phase at the ferrite/austenite grain boundaries.

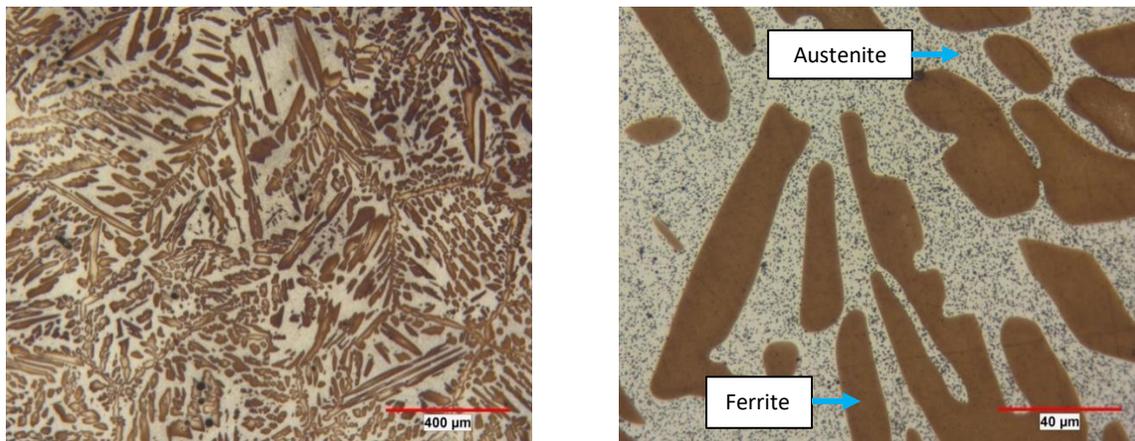


Figure 5. LOM images at two different magnifications showing the microstructure of 2507 sintered at 1365°C for 2hrs in the 95 vol.% N₂/ 5 vol.% H₂ atmosphere then heat treated at 1093°C for 2hrs in a nitrogen atmosphere and water quenched. (etched with Beraha's reagent for duplex SS)

Figure 6 shows the microstructure of the 2507 duplex samples that were sintered in a hydrogen atmosphere and heat treated. In Figure 6a there are grains of ferrite and a secondary phase along the grain boundaries. This sample would have decreased corrosion resistance due to the presence of the secondary phase reducing the effective chromium content. The sample in Figure 6b was heat treated at 1149°C for 4 hours in a nitrogen atmosphere then water quenched. The secondary phase that was along the grain boundaries is now dissolved.

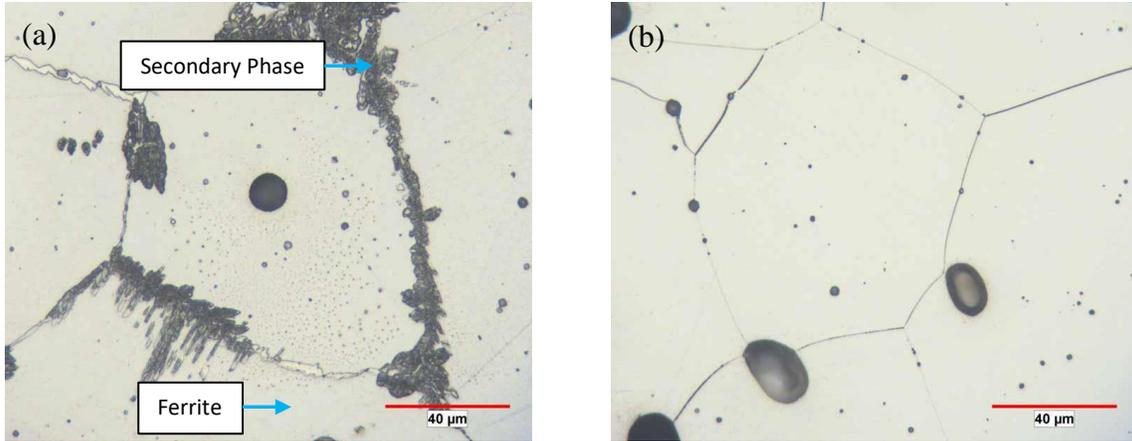


Figure 6. LOM image showing microstructure of (a) 2507 sintered at 1380°C for 2hrs in a hydrogen atmosphere and (b) 2507 sintered and heat treated at 1149°C for 4hrs in a nitrogen atmosphere then water quenched. (etchant Glyceregia)

Mechanical Properties – Nickel Free Stainless Steel

Nickel-free stainless steel binder jet samples were sintered with one set of conditions and also heat treated. Table VII shows the mechanical properties achieved after sintering in a nitrogen atmosphere at 1270°C for 6 hours. A lower temperature and longer hold time than the 316L samples was used for the nickel-free stainless steel because a higher nitrogen level was required for this material and the solubility for nitrogen decreases with increasing temperature and decreasing nitrogen partial pressure.¹¹ With these conditions the samples reached a 7.44 g/cm³ density and 0.49% nitrogen level. The YS and UTS met the wrought minimum specification, but the elongation was slightly lower at 37%. Considering the increased porosity content of the as-sintered binder jet samples the properties are very close to the wrought specification. Performing a solution anneal could further improve the elongation or using hot isostatic pressing.

Table VII. Mechanical properties of nickel-free SS binder jet samples and the wrought specification

Ni-Free SS Material	Density [g/cm ³]	N [%]	0.2%YS [MPa]	UTS [MPa]	Elong. [%]	Apparent Hardness [HRA]
UNS S29225, Annealed	---	0.45-0.55	482 min	827 min	40 min	---
Binder Jet, Sintered (a)	7.44	0.49	496	848	37	60

(a) Sintered at 1270°C for 6hrs in a nitrogen atmosphere

Figure 7 shows the etched microstructures of sintered and heat treated binder jet nickel-free stainless steel. In Figure 7a the lighter areas are austenite and the gray areas are delta ferrite. There is also a secondary phase along the ferrite/austenite grain boundaries. To dissolve the secondary phase, which can deteriorate mechanical properties as well as corrosion resistance, the samples were also solution annealed as shown in Figure 7b.

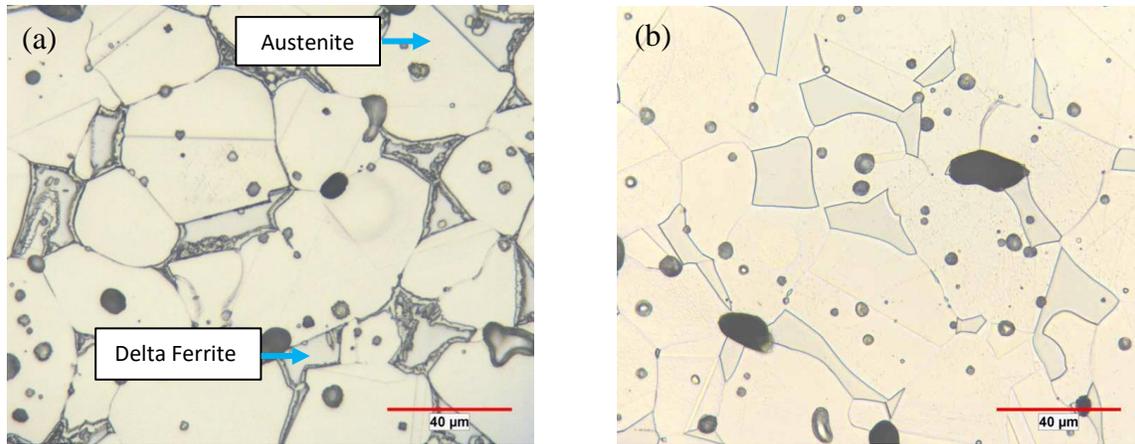


Figure 7. LOM image showing the microstructure of (a) nickel-free SS sintered at 1270°C for 6hrs in a nitrogen atmosphere and (b) nickel-free SS sintered and heat treated at 1093°C for 30mins in a nitrogen atmosphere and water quenched. (etchant Glyceregia)

Delta Ferrite

The presence of delta ferrite could cause the alloy to become slightly magnetic and may be detrimental to the material properties in certain applications. An additional heat treatment was used to try to dissolve the phase in the binder jet 316L and nickel-free stainless steel samples. Both samples were heated to 1010°C in a nitrogen atmosphere and held there for 7 hours followed by water quenching. At this temperature both alloys are in an austenite region at equilibrium. In Figure 8a you can see a reduction in the amount of delta ferrite as compared to the heat treated sample in Figure 2b. Similarly, the delta ferrite has significantly reduced in the sample in Figure 8b as compared to the as-sintered sample in Figure 7a. If the dissolution of delta ferrite is desired tailored heat treatments could be used.

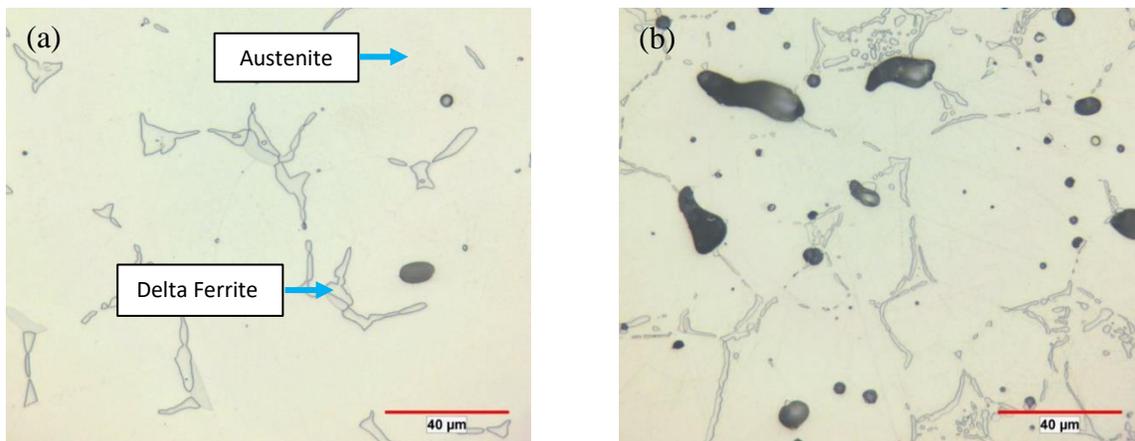


Figure 8. LOM image showing the microstructure of (a) 316L sintered at 1380°C for 2hrs in a hydrogen atmosphere and heat treated at 1010°C for 7hrs in a nitrogen atmosphere and water quenched and (b) nickel-free SS sintered at 1270°C for 6hrs in a nitrogen atmosphere and heat treated at 1010°C for 7hrs in a nitrogen atmosphere and water quenched. (etchant Glyceregia)

Material Property Summary

Table VIII shows the as-sintered properties of the three alloys that achieved the targeted level of nitrogen from the wrought specification and Figure 9 shows the corresponding microstructure. The nickel-free stainless steel and the 2507 duplex achieved twice the YS of the 316L binder jet samples and had increased UTS and hardness. The 316L binder jet samples had the highest elongation due to its high

density. The nickel-free stainless steel was able to achieve a similar microstructure to the 316L sample by its increased nitrogen content as shown in Figure 9a and c. Both microstructures show austenite and delta ferrite with a secondary phase along the austenite/ferrite grain boundaries. The 2507 duplex microstructure also contains a mixture of austenite and ferrite along with a secondary phase. When high strength and corrosion resistance are required 2507 duplex and nickel-free stainless steel could be suitable alternatives to 316L.

Table VIII. Mechanical properties of as-sintered binder jet specimens

Condition	C [%]	N [%]	0.2% YS [MPa]	UTS [MPa]	Elong. [%]	Apparent Hardness [HRA]
316L (a)	0.02	0.004	197	569	49	41
2507 (b)	0.05	0.27	534	780	19	59
Ni-Free SS (c)	0.06	0.49	496	848	37	60

(a) 316L, Sintered at 1380°C for 2hrs in hydrogen atmosphere

(b) 2507, Sintered at 1365°C for 2hrs in 95 vol.% N₂ / 5 vol.% H₂ atmosphere

(c) Ni-Free SS, Sintered at 1270°C for 6hrs in nitrogen atmosphere

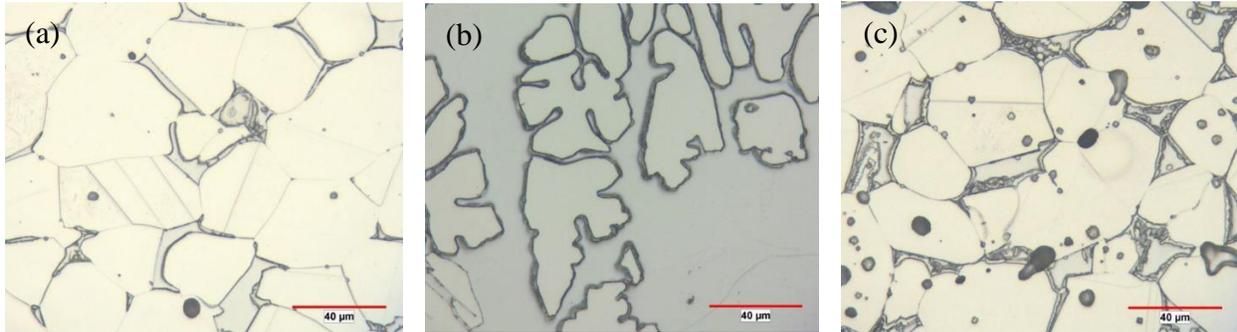


Figure 9. LOM image showing the microstructure of (a) 316L sintered at 1380°C for 2hrs in a hydrogen atmosphere, (b) 2507 duplex sintered at 1365°C for 2hrs in 95 vol.% N₂ / 5 vol.% H₂ and (c) nickel-free SS sintered at 1270°C for 6hrs in a nitrogen atmosphere. (etchant Glyceregia)

CONCLUSIONS

- 316L, 2507 duplex, and nickel-free stainless steel samples produced by binder jet were all able to meet or closely match the wrought mechanical property specification for the corresponding alloy.
- The sintering temperature, time and atmosphere were found to be critical factors in achieving the proper density and nitrogen level in nickel-free stainless steel and 2507 duplex.
- Fast cooling from the sintering temperature may be needed to prevent the precipitation of secondary phases or a solution anneal may be required to dissolve any secondary phases that form during the sintering process to improve the corrosion resistance of the as-sintered binder jet specimens.
- Nickel-free stainless steel and 2507 duplex were shown to be possible alternatives to 316L if lower nickel content is required for binder jet part applications.

FUTURE WORK

To investigate the corrosion resistance of the three stainless steels used in this study binder jet samples will be used in nickel leach testing following the EN 1811 standard. As-sintered samples will be fully submerged in artificial sweat for a 1 week duration at room temperature. The nickel content of the artificial sweat will then be measured using ICP analysis.

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