Paper

High Density Sintered Stainless Steel

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SYNOPSIS

At present, most stainless steel materials are sintered at high temperatures to achieve the desired mechanical, corrosion and high-temperature properties through densification. Although this method of processing achieves the desired properties, the resulting dimensional change can be difficult to control. A potential alternative to reduce the amount of densification required would be stainless steel powders with higher compressibility, which can reach higher green densities at reasonable compaction pressures. The purpose of this paper will be to compare different techniques to attain the desired sintered properties. The mechanical properties and green and sintered densities reached with these techniques will also be discussed.

KEY WORDS

Stainless steel powder, Warm compaction, Corrosion test, Green strength, Dimensional change

1 Introduction

Stainless powder is mostly used to make car components. Some components are required to withstand severe conditions. In order to meet a requirement such as this, a higher density than 7.2 g/cm^3 provides satisfactory durability¹⁻³⁾ against corrossions and oxidation. Elevated sintering temperatures (above 1250°C) are necessary to achieve these kinds of densities.

One way of reaching higher sintered densities, and thus better mechanical properties, is to increase the compressibility of the powder. An effective way of doing so is by warm compaction, a technique⁴⁾ already shown to increase compressibility of stainless steels with about 0.2 g/cm^3 . Another advantage with the warm compaction technique is that, in general, it creates an even density within the component⁵⁾. This indicates a possibility for manufacturing high-density P/M stainless steel components, e.g. exhaust flanges or HEGOS bosses, with close tolerances.

409L is commonly used in the production of exhaust flanges that require a sintered density of $>7.2 \text{ g/cm}^3$. 316L is commonly used in the stainless steel P/M industry, but its field of application could increase further if a sintered density $>7.2 \text{ g/cm}^3$ can be reached in a more effective way than today.

The study of dimensional change of stainless powder may help PM manufactures when considering the tendency

of both material and process.

2 Experimenal Procedure

2.1 Material

The powders used for this study are as-atomised 409L and 316L with a particle size $<150 \mu\text{m}$. The theoretical densities of 409L and 316L are around 7.7 g/cm^3 and 8.0 g/cm^3 respectively. The composition and particle size of these materials are shown in Table 1 and Table 2.

Five different mixes were made, the mixes for conventional compaction contain 1.0% amide wax and 0.2% Lithium stearate as lubricants. The amide wax is an ethylene-bis-stearamide (EBS), also known as Acrawax®C. The other mixes contain a lubricant designed for warm compaction

Table 1 Chemical composition of the base powder.

Material	Cr (%)	Ni (%)	Si (%)	Mn (%)	Nb (%)	C (%)
409L	12.2	0.1	0.8	0.1	0.6	0.01
316L	16.9	12.8	0.8	0.1	0	0.02

Table 2 Particle size of distribution of the base powder.

Material	-45 μm	45-106 μm	+106 μm
409L	32%	54%	14%
316L	49%	47%	4%

in the amount of 1.2 wt%. The mixes were referred to as 409c, 316c (c for conventional compaction) and 409w 316w (w for warm compaction), with an additional mix 316p, as seen in Table 3. 0.5% phosphorous was added to this mix as Fe_3P ($-30\text{ }\mu\text{m}$) to activate the sintering, and make the target density reachable.

2.2 Compaction and sintering

Tensile strength (TS) test bars, and transverse rupture strength (TRS) test bars according to ISO 2740 and ISO 3995 were compacted at 700Mpa by both conventional compaction and warm compaction. In the case of warm compaction, a temperature of $100\text{ }^\circ\text{C}$ was used for both powder and tool.

Those bars were sintered at several temperatures (1280 , 1340 and $1250\text{ }^\circ\text{C}$) for 45 min. in $100\%\text{H}_2$ atmosphere. A sintering temperature of $1280\text{ }^\circ\text{C}$ was used for 409L. To obtain the required density, conventional compaction followed by densification with high temperature sintering is common practice. As compared to ferritic grade 409L austenitic grade 316L does not shrink as much during sintering, and to reach a density of $> 7.2\text{ g/cm}^3$ with conventional compaction very high sintering temperatures are needed ($> 1340\text{ }^\circ\text{C}$).

In order to reach the target density, it was necessary to sinter the conventionally compacted 316c at a temperature of $1340\text{ }^\circ\text{C}$, while a temperature of $1250\text{ }^\circ\text{C}$ was sufficient for both 316p and 316w (see Table 3).

2.3 Evaluations

Chemical composition and particle size of distribution were analysed according to ISO 7625 and 4497 respectively.

Green Density and Green Strength were measured according to ISO 3927 and 3995. Sintered Density was measured according to ISO 2738 and Dimensional Change was evaluated according to ISO 4492. Then Mechanical properties were measured according to ISO 4498 and 2740.

Table 3 Mixes and procedures used.

Mix	Lubricant	Sintering	Target Density (g/cm^3)
409c	1.0% Amide Wax + 0.2% Lithium-stearate	$1280\text{ }^\circ\text{C}$, 45 min, H_2	7.3
409w	1.2% lubricant for warm compaction	$1280\text{ }^\circ\text{C}$, 45 min, H_2	7.3
316c	1.0% Amide Wax + 0.2% Lithium-stearate	$1340\text{ }^\circ\text{C}$, 45 min, H_2	7.2
316w	1.2% lubricant for warm compaction	$1250\text{ }^\circ\text{C}$, 45 min, H_2	7.2
316p	1.0% Amide Wax + 0.5% P	$1250\text{ }^\circ\text{C}$, 45 min, H_2	7.2

3 Results and Discussions

3.1 Green properties

After compaction green density and green strength were measured. The results can be seen in Table 4, and are illustrated in Fig. 1 and Fig. 2.

The use of warm compaction proves beneficial by increasing the as-compressed density with 0.2 g/cm^3 for 409L and 0.3 g/cm^3 for 316L compared to conventional compaction (Fig. 1).

The phosphorous addition reduces the green density somewhat (Fig. 1). The green density of 316L reached a value of over 6.9 g/cm^3 when warm compacted.

Another advantage when using the warm compaction technique is the increased green strength, (Fig. 2) partly due to the increased green density and partly due to the special type of lubricant used in this mix.⁵⁾

Table 4 Green properties of the mixes.

Mix	Pressure (Mpa)	Green Density (g/cm^3)	Green Strength (Mpa)
409c	700	6.63	9.3
409w	700	6.83	16.6
316c	700	6.62	9.9
316w	700	6.94	18.2
316p	700	6.57	11.7

Fig.1 Green Density of 409L and 316L.

Fig.2 Green Strength of 409L and 316L.

In the case of 409L, the green strength is increased from a value of 9.3 MPa to a value over 16.6 MPa. The strength at green state is important because a higher value makes the components less fragile, and thus cracks are not formed as easily. This simplifies the handling of produced components.

The values for 316w demonstrate an almost twice as high green strength compared to conventional compaction. The value of ~ 18 MPa indicates the possibility of performing green machining, a technique that could potentially increase the application field of this material. 316p has a higher green strength than the reference mix (316c). This tendency has been witnessed before, e.g. for phosphorous additions to ASC100.29⁶⁾

3.2 Sintered properties

The results for density and dimensional change are given in Table 5. The values for density were measured on two TS-bars for each material. Dimensional change was measured as an average of 15 TS bars for each.

In the case of 409L, the difference in sintered density between the two investigated materials compacted at room temperature and warm is about 0.1 g/cm^3 as compared to 0.2 g/cm^3 before sintering. This indicates that after a certain density level, when the sintered part approaches the theoretical density, the sintering activity starts to decrease. Since 409w has a higher green density, this effect takes place earlier in the sintering process, resulting in lower dimensional change. Due to this fact, the difference in sintered densities between 409c and 409w is lower than the difference in green density. The results for sintered density and dimensional change are shown in Fig. 3 and 4.

The diagram in Fig. 5 is the result of a separate study using 409L. The measurements were made on TS-bars sintered in a lifting hearth furnace, with the parameters explained under the Fig. 5. It represents the dimensional change needed in order to reach a certain sintered density,

having the sintering temperature as variable.

In the case of 316L, the target density of 7.2 g/cm^3 was surpassed with mixes 316p and 316w. The conventional mix (316c) reached an acceptable value of 7.18 g/cm^3 . From Table 5 and Fig. 3, it can be seen that 316w reached the target density with a margin of 0.04 g/cm^3 , with 90°C lower sintering temperature than 316c and the lowest dimensional change (green to sintered). The phosphorous containing mix, 316p that was compacted and sintered in the same way as 316w demonstrates the highest dimensional change value. Higher dimensional change normally also generates higher dimensional scatter between the parts which normally reduces tolerances.

Fig.3 Sintered Density of 409L and 316L.

Fig.4 Dimensional Change of 409L and 316L.

Table 5 Densities and Dimensional change of mixes.

Mix	Compaction	Pressure (Mpa)	Sintering	Green Density (g/cm³)	Sintered Density (g/cm³)	Dimensional change (%)
409c	Conventional	700	1280 °C, 45 min, H ₂	6.65	7.27	-3.66
409w	Warm	700	1280 °C, 45 min, H ₂	6.83	7.36	-3.13
316c	Conventional	700	1340 °C, 45 min, 100% H ₂	6.62	7.18	-3.06
316w	Warm	700	1250 °C, 45 min, 100% H ₂	6.94	7.24	-1.84
316p	Conventional	700	1250 °C, 45 min, 100% H ₂	6.57	7.22	-3.39

Figure 5. Dimensional change vs Sintered density for 409L.
Pressure= 700MPa
Sintering time= 45min.
Sintering temperatures= 1160, 1250 and 1340 °C
Sintering atmosphere = 100% H₂

Fig.5 Dimensional Change vs Sintered Density for 409L.

From this diagram it can be seen that warm compaction in general demands less dimensional change and reaches higher densities than conventional compaction. The results obtained confirm this trend. In order to get a sufficient tolerance, increase of green density is necessary. Therefore 316L is more suitable for this than 409L at this process condition.

3.3 Mechanical properties of 410L and 316

The results for mechanical properties are given in Table 6. The values presented are an average of 15 measurements.

The mechanical properties of 316L were better than that of 409L. One of the reasons can be explained by increase of sintering activity due to finer powder for 316L than 410L and no reaction with Nitrogen gas. The tensile and yield strength of warm compacted were slightly superior to that of conventional one but the densification (shrinkage) level

Fig.6 Tensile Strength of 409L and 316L.

of the parts were half. 316p had superior tensile and yield strength to the other two mixes. This result is congruent to what is found for low-alloy P/M powders with phosphorous additions at the same sintered density⁶⁾, e.g. ASC100.29. This can be explained by the effect of phosphorous on shrinkage, pore shape and solution strength. The densification level for 316p was the highest. Regarding Young's Modulus, that of 316P was a bit far from Mac Adam's law (1951).

Though there are lots of factors on mechanical properties, more considerations will be necessary in future to improve them more.

3.4 Dimensional stability of SUS409

A set of 500 rings made of SUS409L were compacted at the same conditions using a 45-ton mechanical press, equipped with a slot heater system and electrical heating of the tools, in order to evaluate the dimensional stability when using warm compaction. The dimensions of the rings were the following: inner diameter 25.4 mm, outer diameter 35.1 mm and height 10 mm. 5 rings per 100 compacted were measured and then sintered (a total of 50 rings per mix) in a ceramic belt type furnace with ceramic trays. The sintering condition was 1282 °C for 20 min in an atmosphere of 50% H₂/50% N₂.

The outer diameter was measured as two perpendicular lines. By calculating the difference as an absolute value, a

Table 6 Mechanical properties of mixes.

Mix	Sintered Density (g/cm³)	Hardness (HV10)	Ultimate Tensile Strength (Mpa)	Yield Strength (MPa)	Young's Modulus (Gpa)	Elongation (%)
409c	7.27	101	370	—	151	16.7
409w	7.36	105	397	—	157	17.4
316c	7.18	91	410	171	142	33.7
316w	7.24	94	419	204	148	28.0
316p	7.22	142	549	297	162	28.6

Fig.7-1 Cross section of 316w TS-bar at low magnification.

Fig.7-2 Centre of cross section of 316w TS-bar at higher magnification.

measurement of roundness was noted for each of the rings. The closer the value was to 0, the closer the ring was to perfect circularity. In order to eliminate the effect of the filling direction the green ring were set in random direction before measurements took place. The sintered rings were then measured in this same direction. The results are presented in Table 7 and are an average of 50 rings.

As can be seen, the warm compacted mix achieves a roundness of $12.27 \mu\text{m}$ as compared to $22.48 \mu\text{m}$ for the cold compacted standard mix. This corresponds to a reduction of dimensional scatter to almost half. This tendency can be the same for SUS316L. This shows that the process of warm compaction generates better dimensional stability when using stainless steel powder.

It should be mentioned that the non-complex geometry of the ring resulted in relatively low scattering values. An even density within components⁵⁾ with the warm

Table 7 Roundness stability of mixes.

Material		Roundness	St. dev
409c	Green State (microns)	4.78	4.07
	Sintered State (microns)	22.48	14.69
409w	Green State (microns)	5.78	5.03
	Sintered State (microns)	12.27	9.54

compaction should be better to control the dimensions of complex component too.

4 Conclusions

- (1) The green density of stainless steel powders is increased with $> 0.2 \text{ g/cm}^3$ by the means of warm compaction.
- (2) The green strength of stainless steel powders is increased substantially by the means of warm compaction.
- (3) Warm compaction of stainless steel powders results in higher sintered density with less dimensional change as compared to cold compaction.
- (4) Warm compacted stainless steel powders exhibit a more even porosity distribution, with fewer amounts of large pores.
- (5) Warm compaction gives us possibility to produce more accurate components.
- (6) Austenitic stainless 316L provides less dimensional change than Ferritic stainless 409L.
- (7) Phosphorous additions to 316L activate sintering and increase mechanical properties but induce higher dimensional change as compared to both cold and warm compaction.

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