HIGH DENSITY SINTERED STAINLESS STEEL

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Abstract

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.

Introduction

The automotive industry's increasing interest in using stainless steel P/M parts emphasizes the property requirements for the powder materials used. For many of the produced components, the density required to achieve satisfactory corrosion and high-temperature properties is a minimum of 7.2 g/cm^3, [1] [2]. To achieve these kinds of densities, elevated sintering temperatures (above 2280°F / 1250°C) are necessary.

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 technique already shown to increase compressibility of stainless steels with about 0,2 g/cm^3, [3]. Another advantage with the warm compaction technique is that, in general, it creates an even density within the component, [4]. This indicates a possibility for manufacturing high-density P/M stainless steel components, e.g. exhaust flanges or HEGO bosses, with close tolerances.

The paper is divided in to two studies. In study 1 warm compaction is compared to conventional when using the same process parameters in order to reach high densities. The material used for this study was ferritic stainless steel 409L. Study 2 compares warm compaction to two other methods of reaching a sintered density of 7.2 g/cm^3 with austenitic stainless steel 316L powder.

Study 1: Warm compaction of 409L

The powder used for this study was as-atomised 409L with a particle size <150µm. This material is commonly used in the production of exhaust flanges that require a sintered density of >7,2 g/cm^3. The theoretical density of the material is around 7,7 g/cm^3. To obtain the required density, conventional compaction followed by densification with high temperature sintering is common practice. The composition of the material is shown in table A.

Table A. Chemical composition and particle size of the powder in study 1

Material	Fe (%)	Cr (%)	Ni (%)	Si (%)	Mn (%)	Cb (%)	C (%)	-45 µm	45-106 µm	+106 µm
409L	Bal.	12.2	0.1	0.8	0.1	0.6	0.01	32%	54%	14%

The objective of study 1 was to identify differences with the use of warm compaction instead of conventional compaction, using a given set of process parameters. The compaction pressure was set to 51 Tsi (700 MPa) and the sintering to 2336 ºF (1280ºC) for 45 minutes in a 100% H_2 atmosphere. Two different mixes were made, the first containing 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 second mix contained a lubricant designed for warm compaction in the amount of 1.2wt%. The mixes were referred to as *409c* (c for conventional compaction) and *409w* (w for warm compaction), as seen in table B.

Table B. Mixes used in study 1

Mix	Lubricant	Compaction	Pressure (Mpa/Tsi)	Sintering
409c	1.0% Amide Wax + 0.2%Lithium-stearate	Conventional	700 / 51	2336ºF (1280ºC), 45 min, H_2
409w	1.2% lubricant for warm compaction	Warm	700 / 51	2336ºF (1280ºC), 45 min, H_2

Green properties

Tensile strength (TS) test bars, and transverse rupture strength (TRS) test bars were compacted. In the case of warm compaction a temperature of 212ºF (100ºC) was used for both powder and tool. After compaction green density and green strength were measured. The results can be seen in table C, and are illustrated in figures 1 and 2.

Table C. Green properties of the mixes in study 1

Mix	Compaction	Pressure (Mpa/Tsi)	Green Density (g/cm³)	Green Strength (MPa/Psi)
409c	Conventional	700 / 51	6.63	9.3 / 1349
409w	Warm	700 / 51	6.83	16.6 / 2408

In the case of 409L the use of warm compaction proves beneficial by increasing the as-compressed density with 0.2 g/cm^3 compared to conventional compaction (see figure 1).

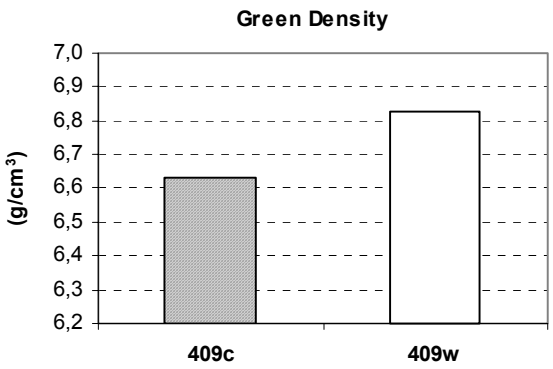


Figure 1. Green density for 409L

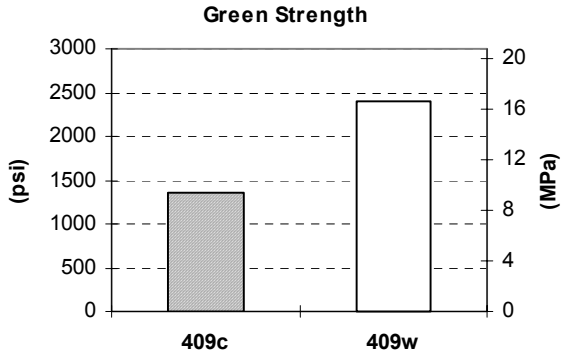


Figure 2. Green strength for 409L

Another advantage when using the warm compaction technique is the increased green strength, [4]. Figure 2 illustrates the case for 409L. From a value of ~1350 psi the green strength is increased to a value over 2408 psi (9.3 to 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.

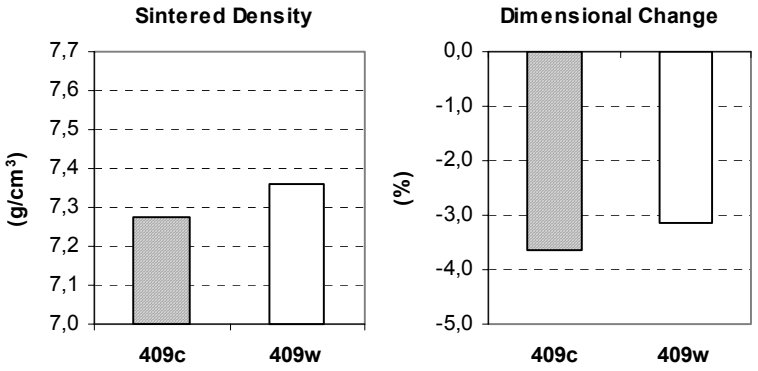
Sintered properties

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

Table D. Densities and Dimensional change of mixes in study 1

Mix	Compaction	Pressure (Mpa/Tsi)	Sintering	Green Density (g/cm^3)	Sintered Density (g/cm^3)	Dimensional change (%)
409c	Conventional	700 / 51	2336ºF (1280ºC), 45 min, H_2	6.65	7.27	-3.66
409w	Warm	700 / 51	2336ºF (1280ºC), 45 min, H_2	6.83	7.36	-3.13

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 cycle, 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 figures 3 and 4.



Figures 3 and 4. Sintered Density and Dimensional change of cold and warm compacted 409L

The diagram in figure 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 figure. It represents the dimensional change needed in order to reach a certain sintered density, having the sintering temperature as variable. 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 in study 1 confirm this trend.

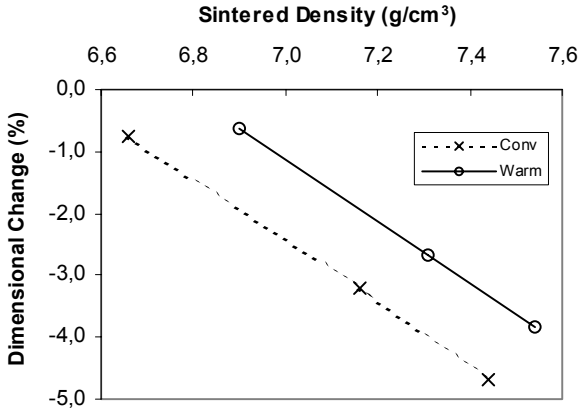


Figure 5. Dimensional change vs Sintered density for 409L.
Pressure= 51 Tsi (700MPa)
Sintering time= 45min.
Sintering temperatures= 2120, 2282 and 2480ºF (1160, 1250 and 1340 ºC)
Sintering atmosphere = 100% H_2

Dimensional stability

To evaluate the dimensional stability when using warm compaction, a set of 500 rings were compacted at 51 Tsi (700MPa) using a 45-ton mechanical press, equipped with a slot heater system and electrical heating of the tools. The dimensions of the rings were the following: inner diameter 1.02 in (25.4 mm), outer diameter 1.40 in (35.1 mm) and height 0.40 in (10 mm). Mix *409c* was conventionally compacted and *409w* at a temperature of 212ºF (100ºC) (both powder and tool). 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 cycle was 2340ºF (1282ºC) for 20min, in an atmosphere of 50%H_2 / 50%N_2.

The outer diameter was measured as two perpendicular lines. By calculating the difference as an absolute value, a measurement of ovality 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 F and are an average of 50 rings.

Table F. Ovality of mixes in study 1

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

Table F demonstrates the ability of the rings to stay round after sintering for the different mixes. As can be seen, the warm compacted mix achieves an ovality of 12.27 µm as compared to 22.48 µm for the cold compacted standard mix. This corresponds to a reduction of dimensional scatter to almost half. This shows that the process of warm compaction generates better dimensional stability when using stainless steel powder 409L.

Figures 8a and 8b demonstrates the dimensional scatter of sintered specimens. The measurement was made on 50 rings of a total of 500. From these diagrams it can be concluded that *409w* demonstrates more dimensional stability since it shows less deviation from the ideal value of 0. It should be mentioned that the non-complex geometry of the ring resulted in relatively low scattering values.

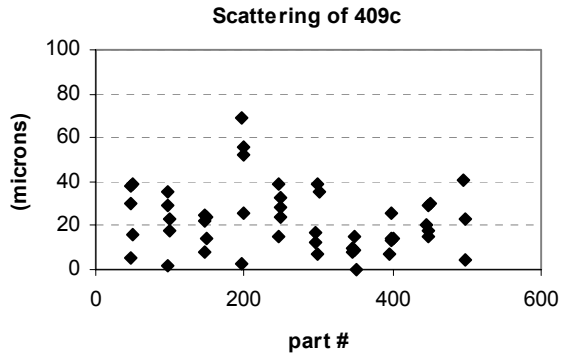


Figure 8a. Scattering of 409c

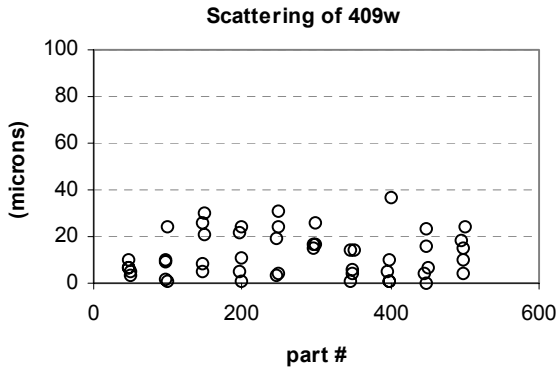


Figure 8b. Scattering of 409w

Study 2: Warm compaction of 316L

The powder used for this study was austenitic stainless steel 316L with a particle size <150µm. This material is commonly used in the stainless steel P/M industry, but its field of application could increase further if a sintered density >7,2 g/cm^3 can be reached in a more effective way than today. The theoretical density of the material is around 8,0 g/cm^3. As compared to ferritic grade 409L this material does not shrink as much during sintering, and to reach a density of >7,2 g/cm^3 with conventional compaction very high sintering temperatures are needed (>2440ºF / 1340ºC). The composition of the material can be found in table G.

Table G. Chemical composition and particle size of the powder in study 2

Material	Fe (%)	Cr (%)	Ni (%)	Si (%)	Mn (%)	Nb (%)	C (%)	-45 µm	45-106 µm	+106 µm
316LHD	Bal.	16.9	12.8	0.8	0.1	0	0.02	49%	47%	4%

The aim of this study was to compare warm compaction with two other methods for reaching a sintered density of >7,2 g/cm^3. The compaction pressure was set to 51 Tsi (700 MPa).
Three different mixes were used in this study, as can be seen in table H.

Table H. Mixes used in study 2

Mix	Lubricant / *Addition*	Compaction	Pressure (Mpa/Tsi)	Sintering	Target Density (g/cm^3)
316c	1.0% Amide Wax + 0.2%Lithium-stearate	Conventional	700 / 51	2444ºF (1340ºC), 45 min, H_2	7.2
316p	1.0% Amide Wax + *0.5% P*	Conventional	700 / 51	2282ºF (1250ºC), 45 min, H_2	7.2
316w	1.2% lubricant for warm compaction	Warm	700 / 51	2282ºF (1250ºC), 45 min, H_2	7.2

As in study 1, the mixes were referred to as *316c* (c for conventional compaction) and *316w* (w for warm compaction), with an additional mix *316p*. 0.5% phosphorous was added to this mix as Fe_3P (-30µm) to activate the sintering, and make the target density reachable.

In order to reach the target density, it was necessary to sinter the conventionally compacted *316c* at a temperature of 2444ºF (1340ºC), while a temperature of 2282ºF (1250ºC) was sufficient for the others.

Green properties

Tensile strength (TS) test bars, and transverse rupture strength (TRS) test bars were compacted for all mixes. In the case of warm compaction a temperature of 212ºF (100ºC) was used for both powder and tool. After compaction green density and green strength were measured. The results can be seen in table I, and are illustrated in figures 9 and 10.

Table I. Green properties of the mixes in study 2

Mix	Compaction	Pressure (Mpa/Tsi)	Green Density (g/cm^3)	Green Strength (MPa/Psi)
316c	Conventional	700 / 51	6.62	9.9 / 1436
316p	Conventional	700 / 51	6.57	11.7 / 1697
316w	Warm	700 / 51	6.94	18.2 / 2640

The green density of 316L reached a value of over 6,9 g/cm^3 when warm compacted. This shows that an increase of ~0,3 g/cm^3 of green density is possible for this material when using this technique. The phosphorous addition reduces the green density somewhat, (see figure 9). As mentioned in study 1, a general advantage of warm compaction is increased green strength, partly due to the increased green density and partly due to the special type of lubricant used in this mix, [4]. A higher green strength value simplifies the handling of components. Figure 9 shows the gain in green strength for stainless steel 316L. The values for *316w* demonstrate an almost twice as high green strength compared to conventional compaction. The value of 2640 psi (~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 ASC100.29, [5].

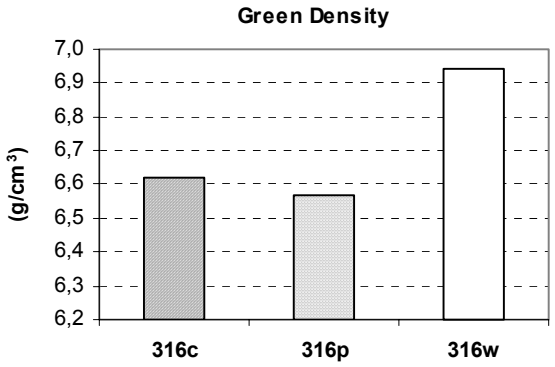


Figure 9. Green density for 316L

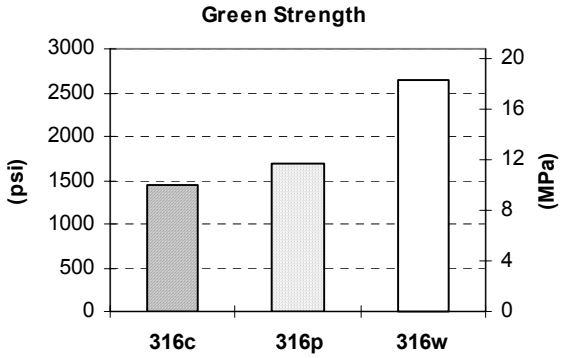


Figure 10. Green strength for 316L

Sintered properties

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

Table J. Sintered density of mixes

Mix	Compaction	Green Density (g/cm^3)	Sintering	Target Density (g/cm^3)	Sintered Density (g/cm^3)	Dimensional change (%)
316c	Conventional	6.62	2444ºF (1340ºC), 45 min, 100% H_2	7.2	7.18	-3.06
316p	Conventional	6.57	2282ºF (1250ºC), 45 min, 100% H_2	7.2	7.22	-3.39
316w	Warm	6.94	2282ºF (1250ºC), 45 min, 100% H_2	7.2	7.24	-1.84

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 J and figure 11, it can be seen that *316w* reached the target density with a margin of 0,04 g/cm^3, with 160ºF 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.

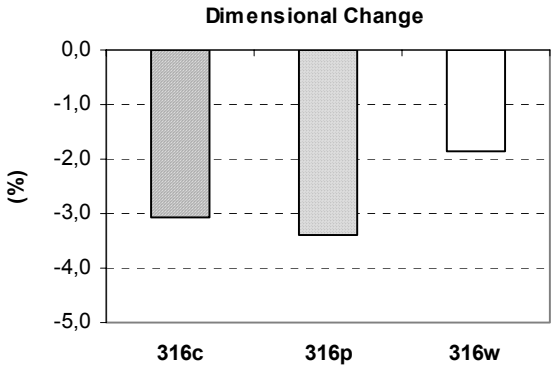


Figure 11. Dimensional change. The values are an average of 15 TS bars.

Porosity distribution

Another general advantage with warm compaction is improved porosity distribution at a given density, [4]. Randomly picked TS-bars were chosen from each of the mixes, and metallographic images were taken to confirm this. Figures 12-17 demonstrate the pore morphology of the TS-bars.

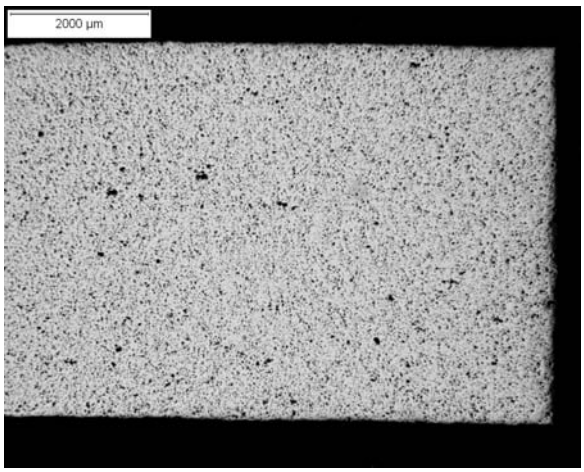


Figure 12. Cross section of *316c* TS-bar at low magnification.

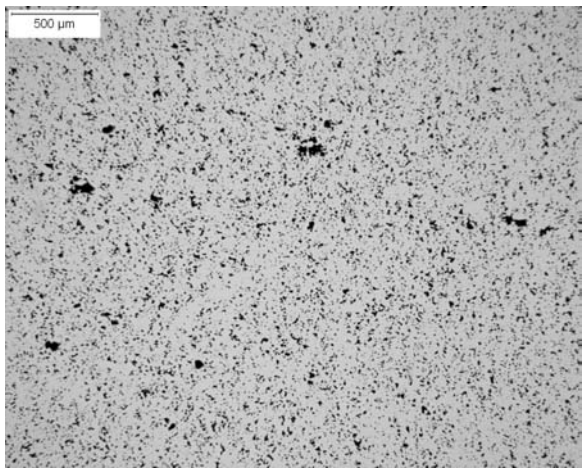


Figure 13. Centre of cross section of *316c* TS-bar at higher magnification.

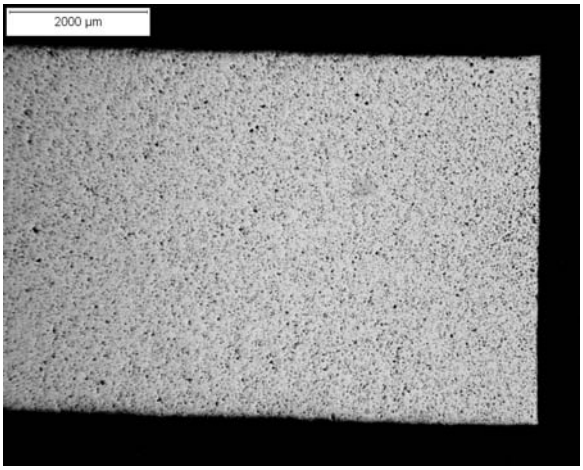


Figure 14. Cross section of *316w* TS-bar at low magnification.

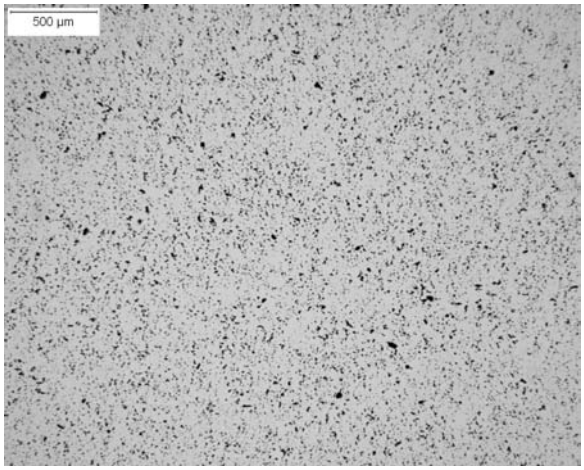


Figure 15. Centre of cross section of *316w* TS-bar at higher magnification.

Images 12 to 15 demonstrate that warm compaction, even at these small dimensions, results in a more even porosity distribution than conventional. The large pores found in the *316c* sample are primarily due to large particles being present in the lubricant used and are not visible for *316w*. This confirms what have been found for low-alloyed PM steels, [4]



Figure 16. Cross section of *316p* TS-bar at low magnification.

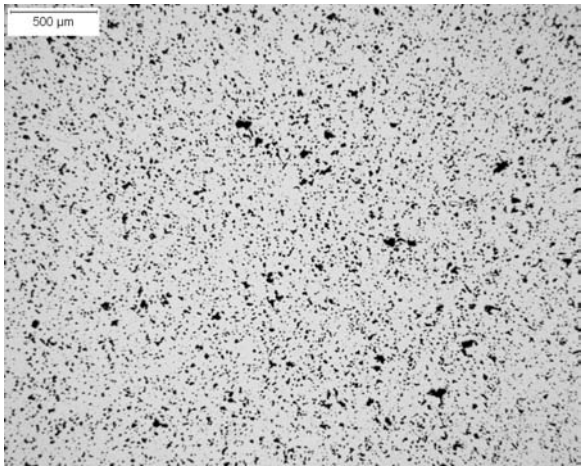


Figure 17. Centre of cross section of *316p* TS-bar at higher magnification.

In figures 16 and 17 it can be seen that the phosphorous containing mix exhibits a porosity distribution with some large pores similar to those of the *316c* samples. Pores are randomly distributed over the cross section. However, the phosphorous addition results in closed porosity instead of interconnected, as well as rounded pores. This is advantageous both from strength and corrosion resistance point of view, [6].

Mechanical properties

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

Table K. Mechanical properties of mixes in study 2

Mix	Sintered Density (g/cm^3)	Hardness (HV10)	Ultimate Tensile Strength (MPa / ksi)	Yield Strength (MPa / ksi)	Young's Modulus (GPa / Msi)	Elongation (%)
316c	7.18	91	410 / 59.5	171 / 24.8	142 / 20.6	33.7
316p	7.22	142	549 / 79.6	297 / 43.1	162 / 23.5	28.6
316w	7.24	94	419 / 60.8	204 / 29.6	148 / 21.5	28.0

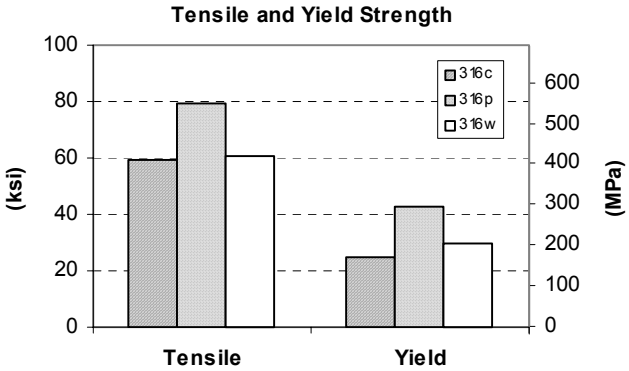


Figure 18. Tensile and Yield strength for 316L

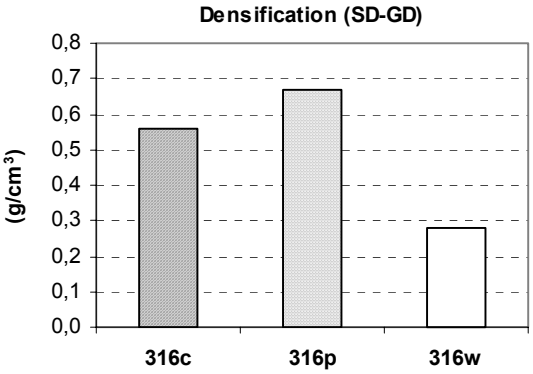


Figure 19. Densification during sintering for the three investigated materials

The tensile and yield strength of *316w* were slightly superior to that of *316c* but the densification (shrinkage) level 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 density, e.g. ASC100.29, [5]. The densification level for *316p* was the highest.

Conclusions

- The green density of stainless steel powders is increased with >0.2 g/cm^3 by the means of warm compaction
- The green strength of stainless steel powders is increased substantially by the means of warm compaction
- Warm compaction of stainless steel powders results in higher sintered density with less dimensional change as compared to cold compaction
- Warm compacted stainless steel powders exhibit a more even porosity distribution, with fewer amounts of large pores
- Phosphorous additions to 316L activate sintering and increase mechanical properties but induce higher dimensional change as compared to both cold and warm compaction

References

1. A. Bergkvist, ”Warm compaction of stainless steel powders”, *Powder Metallurgy World Congress*, Granada, 1998.

2. S. Shah, J.R. Mcmillen, P.K. Samal and E. Klar, “Development of powder metal stainless steel materials for exhaust system applications”, 1998 Society of automotive engineers, Inc.

3. R. Canto Leyton and O.Andersson , “High density sintered stainless steel with close tolerances”, *World Congress on powder metallurgy & particulate materials*, Orlando, 2002.

4. U. Engström, “High performance PM materials by warm compaction of Densmix powders”, *Powder Metallurgy World Congress*, Vancouver, 1999.

5. “Höganäs Iron and steel powders for sintered components”, Höganäs, 2002.

6. A. Molinari, L. Fedrizzi and D. Fax, “How phosphorous affects the mechanical properties and corrosion resistance of P/M stainless steel*”, International journal of powder metallurgy,* volume 38, No. 1, 2002.