FERRITIC STAINLESS STEEL FOR HIGH DENSITY APPLICATIONS

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Abstract

At present, high sintering temperatures are essential in order to meet the density requirements of ferritic stainless steels intended for high performance applications. Use of high sintering temperatures leads to large dimensional changes, making close tolerances more difficult to achieve. By increasing compressibility of the powder, less shrinkage is necessary to meet the density requirements and closer tolerances can be achieved.

This paper will give a general overview of advantages and disadvantages of different process routes available to reach a final density of 7,3 g/cm^3 with ferritic stainless steel 409L. One of the processes discussed here is warm compaction, a technique already proven to increase compressibility of ferritic stainless steels by about 0,2 g/cm^3. Green and sintered properties, including tensile test data, are compared.

Introduction

Ferritic P/M stainless steels, and grade 409L in particular, have had an increased success in the automotive applications in recent years. 409L is currently used to a large extent in the manufacturing of flanges for the stainless steel exhaust systems of U. S. made automobiles. Interest for this application in Europe has not been so strong, but appears to be steadily increasing. The American auto industry has been working with ferritic P/M stainless steels for nearly a decade and has gained a significant level of experience regarding the requirements of the application, and on how to meet them. The most important requirements for this component are density and weldability. The weldability of a ferritic stainless steel is insured by keeping the carbon levels as low as possible, and can be further insured by including a so-called stabiliser, [2]. In the case of P/M 409L the stabiliser selected is Niobium, Nb (referred to as Columbium, Cb in the US).

Because of the relatively low compressibility of these types of powders, the density requirement is met by imparting a high degree of shrinkage during sintering, which in turn requires very high sintering temperatures (>1300ºC). Process control becomes very critical in order to insure that precise dimensions are achieved after densification. To insure a high rate of shrinkage, the manufacturers of flanges work with powders containing large amounts of fine particles (around 50% < 45µm). The high amounts of fine particles do give greater shrinkage during sintering, owing to their higher specific surface area, but this approach does affect other properties negatively such as flow rate, apparent density and compressibility.

The sintered density (SD) attainable of a component is determined by three factors: apparent density (AD), density increase from compaction (ΔD_c) and the densification during sintering (ΔD_s). These simplified correlations can be written as:

$$GD = AD + \Delta D_c$$
$$SD = GD + \Delta D_s$$

For a component such as the exhaust flange the required minimum sintered density is 7,2 g/cm^3, [1], with 7.3 g/cm^3 being the most preferred density in the industry. This paper focuses on how to reach this level of density with ferritic stainless steel 409L. Typically, depending on particle size, lubricant type and part geometry, at a compaction pressure of 600MPa the green density (GD) for this material falls in the range of 6,42-6,52 g/cm^3. This implies that a densification of ~0,8 g/cm^3 needs to be achieved in sintering in order to reach the target sintered density of 7,3 g/cm^3. However, by employing warm compaction the amount of densification needed can be decreased substantially. Warm compaction has been proven to increase the green density by ~0,2 g/cm^3, [3], and will therefore decrease the amount of densification needed during sintering to reach 7,3 g/cm^3 with the same amount. The second advantage of warm compaction is a significant increase in green strength, [3], which can simplify the handling of these components substantially.

This paper discusses differences between cold and warm compaction when processed under different sintering temperatures, keeping in mind a target sintered density of 7,3 g/cm^3. Other properties, such as sintered and mechanical properties are also presented and compared.

Powder properties

As mentioned in the introduction, the properties of the powder, apparent density in particular, will have significant effects on the sintered density attained. Apparent density is in turn influenced by the particle size distribution, particle shape and the type and amount of lubricant. Another important parameter for manufacturing purposes is the flow rate, which determines how fast a given volume of die cavity can be filled with the powder, and therefore influences the rate of production. When attempting to perform warm compaction it is therefore desirable not to alter any of the powder properties adversely. In order to fulfil this requirement a special type of warm compaction lubricant has been developed which produces similar powder properties at warm compaction temperatures (100-120 ºC) as most regular lubricants do at room temperature.

Table A. Chemical composition and powder properties (unlubed)

Material	Fe (%)	Cr (%)	Ni (%)	Si (%)	Mn (%)	Nb (%)	C (%)	-45 (µm)	45-105 (µm)	+105 (µm)	Apparent Density (g/cm^3)	Flow Rate* (s/50g)
409L**	Bal.	11.4	0.06	0.95	0.22	0.54	0.03	43.2	44.5	12.3	2.94	25.8

*) Measured with Hall flow meter
**) Annealed powder

Two mixes were made with the powder shown in Table A. One mix was prepared for use as the reference, while the other was prepared as a mix for warm compaction. The apparent density and flow rate of these two mixes are shown in Table B.

Table B. Apparent density and flow rate

Mix	Lubricant	Apparent Density (g/cm^3)	Flow Rate* (s/50g)
409L-c	1.0% Amide Wax + 0.2%Lithium-Stearate	2.81	33.5
409L-w	1.2% lubricant for warm compaction	3.03	34.6

*) Measured with Hall flow meter

The flow rate and apparent density presented in Table B were measured at room temperature for the c-mix and at 100ºC (212ºF) for the w-mix. At this higher temperature, the mix designed for warm compaction demonstrates a flow rate comparable to that of the reference mix at room temperature, coupled with a higher apparent density. This implies that the filling characteristics during warm compaction of these mix are similar to those exhibited by the reference mix during cold compaction.

Green Properties

There are two important properties of the powder at green state. These are the green density and the green strength. The first one will determine the amount of densification needed to reach our target density, and the second is important for safe handling of green components. Low green strength leads to production of fragile components, which will require greater care in handling, as compared to components with high green strength. To simplify what influences the green density we can write:

$$GD = AD + \Delta D_c$$

Apparent density will thus influence the green density of the component. This parameter is often dictated by the tool design parameters. Different manufacturers have preferences for different AD:s based on the complexity of the component. The second parameter, the density increase due to compaction, is a variable and it depends on the compaction pressure and the compressibility (softness) of the powder. For a given powder compaction using higher pressures is the primary means of increasing green density. There are two means of enhancing the compressibility of the powder. These two methods may affect both parameters leading to a higher green density. The fist method is called softening annealing. As-atomised ferritic stainless steels particles are in a state similar to that of work hardened materials and therefore they can be softened by annealing. This process lowers the microhardness of the powder particles, which leads to higher green density. Typically, compressibility of annealed powder is about 0.1 g/cm^3 higher than that of as-atomised powder. The second method is the warm compaction. Since this method requires a different type of lubricant (see "powder properties"), the apparent density is also influenced. Because of the decreasing yield strength of the powder with increased temperature, the powder becomes easier to compact. The difference in green density between a warm compacted powder and a cold compacted one is around 0,2 g/cm^3. Figure 1 demonstrates the difference between the powders in Table B.

The steeper slope of the line representing the warm compacted mix in figure 1 also indicates that the gain in green density is higher for higher compaction pressures. This indicates that warm compacted mixes have a slightly different compressibility curve than cold compacted mixes. It is also noted that warm compaction at 600 MPa is equivalent to cold compaction at 800 MPa from green density point of view.

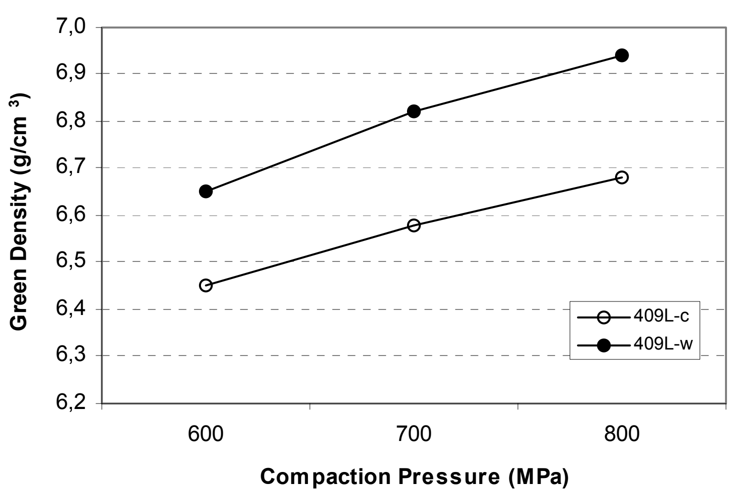


Figure 1. Green Density for a warm and cold compacted mix at different pressures

With this increase in green density it is possible to lower the shrinkage produced during sintering to reach the required sintered density. In practice this would mean that a lower sintering temperature, and/or shorter sintering time could be used. Alternately, the same green density can be targeted using less compaction pressure while maintaining the same sintering cycle.

The green strength is also positively influenced by warm compaction. Green strength is primarily dependent on particle shape: the rounder the particles, the lower the green strength. The powders listed in Table B have good green strength under cold compaction conditions, and when warm compacted the values are exceptionally good. However, if a powder has a marginal green strength under cold compaction conditions, warm compaction can lead to a satisfactory green strength.

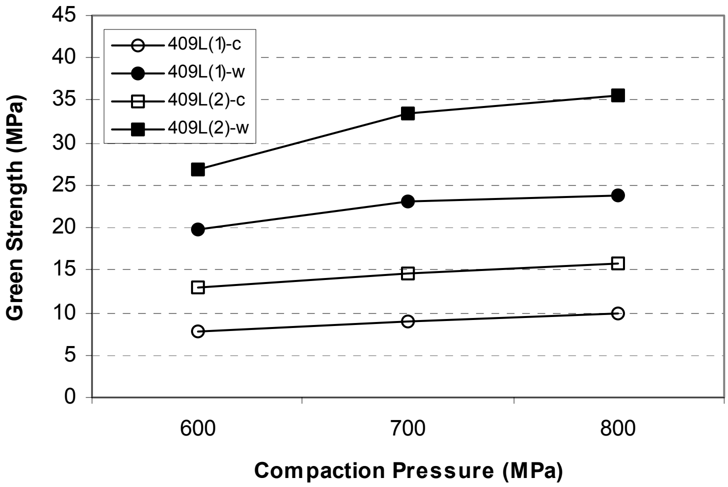


Figure 2. Comparison between two cold and warm compacted lots of 409L with different green strengths.

Figure 2 demonstrates the behaviour discussed above. The powders listed in Table B, denoted 409L(2) in the figure, are compared to a lot that was produced using different atomising parameters, resulting in different particle shapes, and thus different cold compacted green strength. This lot, denoted 409L(1), had a green strength in the range of 7 to 11 MPa, while 409L(2) had a green strength in the range of 12 to16 MPa. Warm compaction increased the green strength substantially in both cases (by more than double). It is also noted that the higher the cold compacted green strength of a powder, the higher is its warm compacted.

Sintered properties

Depending on the green density reached, a suitable sintering cycle has to be chosen in order to reach the target sintered density of 7,3 g/cm^3. However, the green density reached is not the only parameter, which determines the final sintered density. As discussed earlier:

$$SD = GD + \Delta D_s$$

The last parameter represents the shrinkage, or densification during sintering. This is affected by several factors such as the sintering temperature, atmosphere and time. Another important factor is the particle size distribution. Fine particles (< 45µm) activate the sintering process owing to their larger specific surface, which promotes higher densification. This means that two powders with the same green density will only show the same shrinkage if the particle sizes are the same, or at least very similar.

When using the same compaction and sintering parameters, warm compacted powders produce lower shrinkage compared to cold compacted powders. This is mainly due to the fact that the driving force for sintering becomes less, as the density of the part gets closer to the full theoretical density. Hence parts having higher green density shrink less compared to parts having lower density under similar sintering conditions. Since warm compacted parts have a higher green density, they undergo a smaller change in density during sintering, as compared to cold compacted parts.

Standard tensile test (TS)-bars were made from the as-atomised 409L powder described in Table C by compacting at 600, 700 and 800 MPa. Warm compaction was carried out by keeping powder and tool temperature at 110ºC (230ºF). These bars were then sintered in pure hydrogen at three different temperatures: 1160ºC, 1250ºC and 1340ºC. Sintering time was 45 minutes in all cases.

Table C. Chemical composition and powder properties (unlubed)

Material	Fe (%)	Cr (%)	Ni (%)	Si (%)	Mn (%)	Nb (%)	C (%)	-45 (µm)	45-105 (µm)	+105 (µm)	Apparent Density (g/cm^3)	Flow Rate* (s/50g)
409L**	Bal.	11.3	0.1	1.0	0.1	0.5	0.02	40%	49%	11%	2.76	27.1

*) Measured with Hall flow meter
**) As-atomised powder

The same types of mixes as those mentioned in section "Green Properties" were prepared with this lot of 409L powder. The green densities achieved with these mixes are shown in figure 3.

As can be seen in figure 3, the green densities achieved with this powder are very similar to those presented in figure 1. This might seem a little odd, since the powder in figure 1 is annealed while the powder in figure 3 is not. This is due to these two powders having different particle shapes, and thus different as-atomised green densities (the powder in figure 3 had a higher value). If the powder in Table C had been annealed, the green density values in figure 3 would have been around 0,1 g/cm^3 higher.

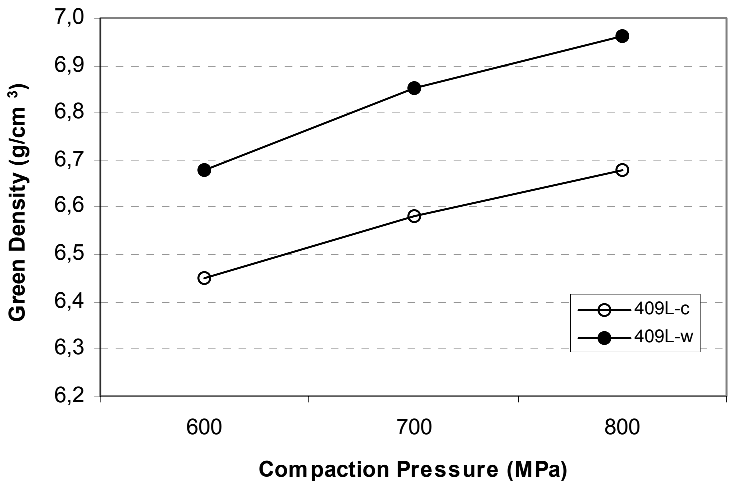


Figure 3. Green Density for warm and cold compacted 409L at different pressures

Since the powders have almost identical green densities in cold compaction, the warm compacted green densities are expected to be similar also. In both cases the green density difference between cold and warm compaction is around 0,2 g/cm^3 at 600 MPa, and it increases slightly at higher compaction pressures (se figure 1 and 3).

As mentioned earlier, when using the same sintering parameters, the difference between sintered densities will be lower than the differences in the green density. This difference becomes smaller and smaller as sintering temperature is increased. This behaviour is shown in figure 4, representing results with sintered as-atomised 409L material from Table C.

Figure 4 provides the opportunity to compare sintered densities achieved with the various process parameters employed here. From this figure, it can be noted that the target of 7,3 g/cm^3 with 409L can be reached by using warm compaction and sintering in pure hydrogen at 1250ºC for 45 minutes for all three compaction pressures. The same sintering parameters when combined with cold compaction will lead to a sintered density of only 7,16 g/cm^3.

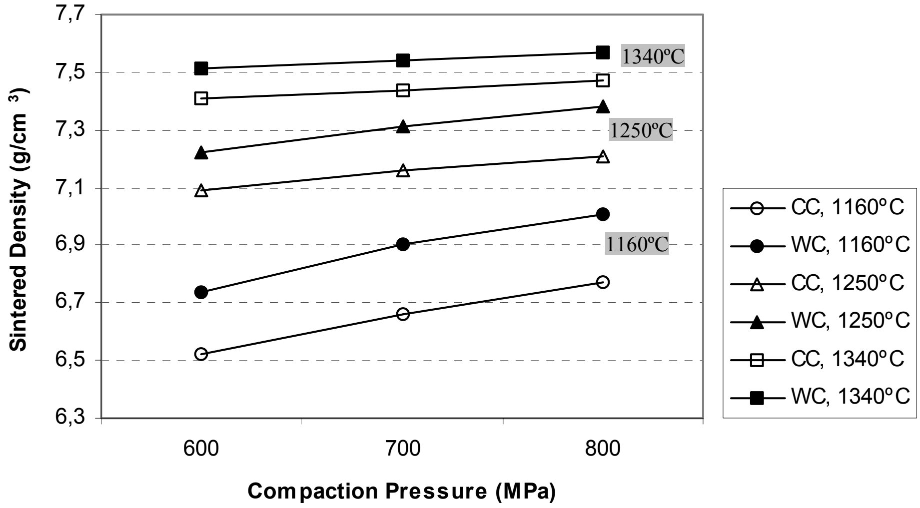


Figure 4. Sintered density for warm (WC) and cold compacted (CC) 409L at different pressures and sintering temperatures

In figure 5, for the same powder, sintered density has been plotted as a function of sintering temperature This figure indicates that to reach 7,3 g/cm^3, with cold compaction using 600 MPa as compaction pressure, the sintering temperature needed will be somewhat higher than 1300ºC. However with warm compaction, using a compaction pressure of 700 MPa, this temperature can be lowered to 1250ºC.

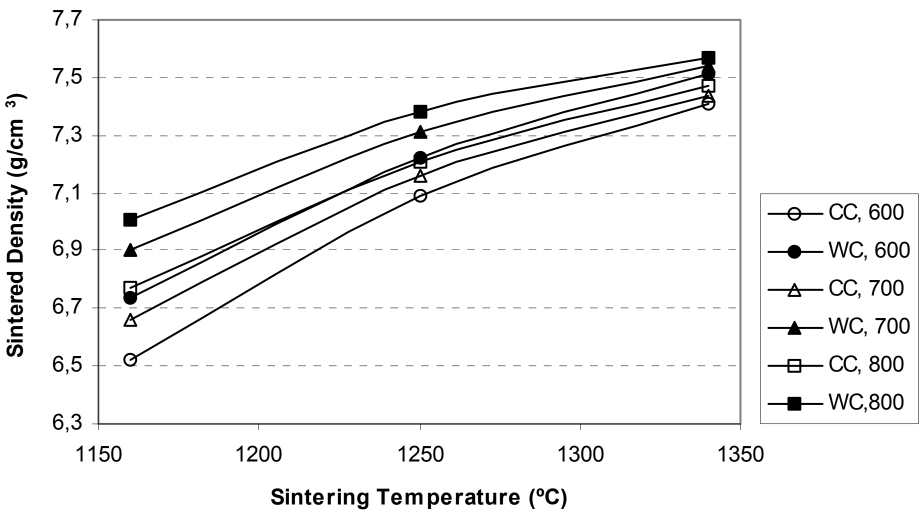
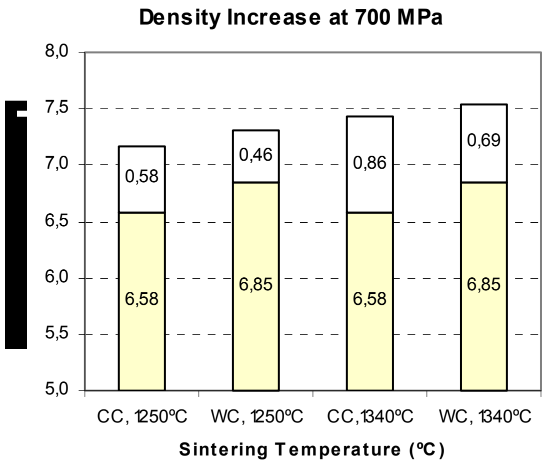
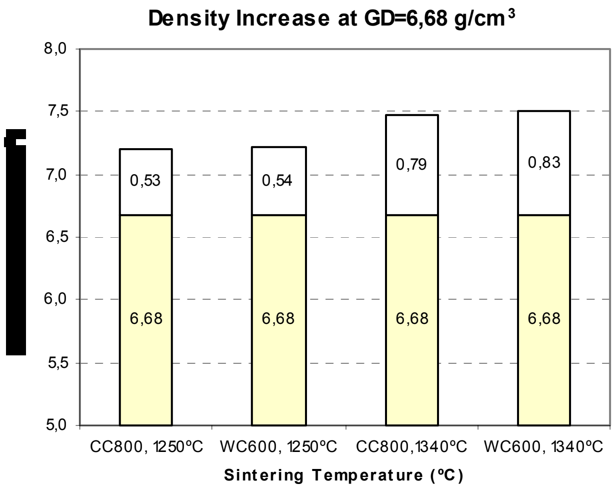


Figure 5. Sintered density for warm (WC) and cold compacted (CC) 409L at different sintering temperatures and pressures

Another interesting aspect in figure 5 is the similarity between the curves for cold compaction at 800 MPa and warm compaction at 600 MPa. These curves are almost overlapping, indicating that if using the same sintering cycle, the compaction pressure can be lowered with 200 MPa when the warm compaction route is chosen. Warm compaction at 800 MPa permits a sintering temperature of around 1230ºC, while using the same compaction pressure the cold compacted route would require a sintering temperature of around 1275ºC.





Figures 6 and 7. On the left, the densification due to sintering is compared for cold and warm compacted 409L at a 700 MPa compacting pressure. On the right this same property is compared but for a green density of 6,68 g/cm^3.

Figures 6 and 7 show the increase in density due to sintering. As may be seen in figure 6 (on the left), under a given set of compaction and sintering parameters warm compaction leads to higher green and sintered densities, combined with a reduced densification (0,58 for cold compaction when sintered at 1250ºC compared to 0,46 for warm compaction). The figure on the right (figure 7) compares densification for a specific green density. The green density of 6,68 g/cm^3 was reached with cold compaction at 800 MPa, while the same was reached with warm compaction at 600 MPa. This figure demonstrates that the shrinkage during sintering for parts with the same green density are quite similar, with a slightly higher shrinkage produced in parts made by warm compaction. This could be due to the more intimate particle-to-particle contacts and stronger bonds, as indicated by the increased green strength obtained with this process.

Mechanical Properties

Mechanical properties are preferably compared with samples that have been sintered at the same time, since some properties are sensitive to any differences in the sintering cycle. Keeping this in mind the comparison in mechanical properties was made between two samples both of which were sintered at 1250ºC and had sintered densities close to 7,3 g/cm^3 . These samples were the cold compacted samples of the powder in Table C at 800 MPa with a sintered density of 7,21 g/cm^3 and the warm compacted samples at 700 MPa with a density of 7,31 g/cm^3.

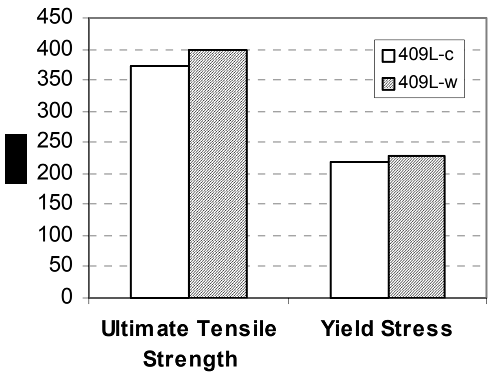


Figure 8. Ultimate tensile strength and yield strength for 409L warm and cold compacted to 7,21 and 7,31 g/cm^3 respectively.

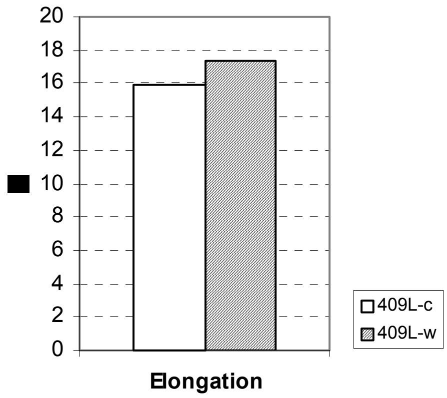


Figure 9. Elongation for 409L warm and cold compacted to 7,21 and 7,31 g/cm^3 respectively.

Figure 8 and 9 demonstrate better mechanical properties achieved with warm compaction. This is primarily due to the difference in sintered density. This indicates that warm compaction does not adversely affect the mechanical properties, when sintered to the same density.

Discussion

Warm compaction provides easier way of reaching a sintered density of 7,3 g/cm^3 with ferritic stainless steel 409L, following from enhancement of green density by around 0,2 g/cm^3. This increase in green density opens up several possibilities. One is to lower the compaction pressure and still reach a desired density, or to reach green densities not reachable with cold compaction because of the pressure limitations of the press. Another option is to decrease the densification during sintering to reach the required density. This is of much commercial interest since high shrinkage generally makes dimensional control difficult to achieve. Another advantage of warm compaction is the increased green strength, which can minimize damage of green components from handling during manufacture.

Using warm compaction, a density of 7,3 g/cm^3 is attainable by compacting at 700 MPa and sintering at 1250ºC in pure hydrogen for 45 minutes. Using the same pressure with cold compaction one would require a sintering temperature of around 1300ºC, which will also result in significantly more shrinkage. Another way of looking at the benefits of warm compaction is that results obtained from warm compaction at 600 MPa are very similar to those obtained from cold compaction at 800Mpa - both involving a sintering temperature of around 1275ºC to reach 7,3 g/cm^3.

Sintering parameters and green density are not the only critical factors responsible for attainment of the final sintered properties. Other important parameters are the microhardness of the particles (annealed or as-atomised powders), particle shape (roundness), particle sizes distribution (amount of fines), and impurities (carbon, oxygen, nitrogen content).

All of these factors will affect properties such as apparent density, flow rate, green density, green strength, shrinkage and dimensional tolerance. Some of these factors will then go on to affect the sintered density attained. Many of these interact with each other. It is therefore clear that there are many different process routes to reach a given sintered density. The most common one at present is to utilize high sintering temperature and use powders with a high amount of fine particles. This will lead to the desired density, but at the cost of high shrinkage. Warm compaction on the other hand, offers higher densities, without the disadvantage of high shrinkage. As a process it also offers a means to improve the green properties of 409L, in addition to achieving improved sintering behaviour. The disadvantage of warm compaction process is that the powder and the tooling of the press have to be heated to temperatures around 100-120ºC, which requires certain modifications and investments.

Conclusions

- Green strength of the compact is more than doubled with warm compaction.
- If using the same sintering cycle, the compaction pressure can be lowered with 200 MPa when the warm compaction route is chosen.
- By utilizing warm compaction, a density of 7,3 g/cm^3 is attainable with 409L with 700 MPa compaction pressure and a sintering temperature of 1250ºC.
- Under a given set of compaction and sintering parameters, warm compaction results in higher green and sintered densities, and is associated with smaller degree of shrinkage.
- Mechanical properties are density dependent. Therefore, for similar sintered densities, cold and warm compaction yield similar mechanical properties.

References

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