# KEY PARAMETERS FOR WARM COMPACTION OF HIGH DENSITY MATERIALS

by

S. St-Laurent and F. Chagnon
Quebec Metal Powders Limited

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**ABSTRACT**

The mechanical properties of high performance P/M parts depend heavily on material composition and the density reached during processing. Warm compaction technique, which consists of pressing preheated powder in a heated die [1], is known to favour densification of parts, leading to an improvement of sintered properties [2,3]. Warm compaction requires powder mixes with specific physical characteristics to be adequately processed in the temperature range involved in warm pressing. In particular, powder mixes should provide good flowability and good lubrication of the die walls in order to reduce ejection forces.

Behaviour and properties of binder and non-binder treated materials processed by the warm compaction technique were investigated both on lab and industrial scales. Specific test programs on a production press were conducted to identify and quantify the effects of different key production parameters such as the compaction pressure, the powder and die temperatures, the production rate and the part size on green and sintered characteristics and ejection forces of parts. The warm compaction process capability is discussed in terms of stability of powder flow rate and apparent density, compacting pressure and temperature as well as weight and density of pressed parts.

**INTRODUCTION**

For numerous P/M applications, high sintered densities, typically over 7.3 g/cm$^{3,}$ are needed in order to achieve high mechanical strength and toughness. Such high densities are difficult to reach using standard compaction and sintering conditions. The warm compaction technique, which consists in pressing preheated powder mix in a heated die [1], is known to increase green density and, in turn, sintered density [2,3]. Final sintered density ranging typically between 7.25 and 7.45 g/cm$^3$ can be achieved by warm compaction with a single compaction/single sintering treatment by using a compacting pressure and a sintering temperature of 50 tsi (690 MPa) and 1120 °C respectively [3,4]. Still higher densities could even be achieved by using high temperature sintering [5].

Powder mixes used for warm pressing should be able to withstand the temperature involved during the compaction cycle. In particular, the lubricant/binder system should provide good flowability and stability (robustness) at the compacting temperature and should enable a target density on a production scale. The density reached on a production press with more complex and heavier parts could be quite different from that reached with small specimens on a lab press. In particular, differences in the compaction processing between a hydraulic lab press and a mechanical production press such as the compaction rate should make a large difference in the powder response to the compacting temperature and pressure. For these reasons, trials on a production press more closely reflecting the day-to-day compaction conditions in the P/M industry are necessary for a proper and more efficient materials development and scale-up from laboratory to production for warm compaction. They are also of key importance in the optimization of the compaction conditions to meet the part producer's specifications and needs such as density, surface finish and part-to-part consistency.

This study is mainly focused on results of warm compaction trials conducted on a production press. The impact of processing parameters such as the powder temperature, compacting pressure and part size on green and sintered properties and process capability and robustness of a binder treated mix containing a high melting point lubricant suitable for warm compaction are presented and discussed.

## EXPERIMENTAL PROCEDURE

### Base Materials and Characterization

Regular and binder treated mixes were compacted on a lab scale press and on a production press [6,7]. Different binder/lubricant systems and lubricant content were investigated. Results presented in this paper were obtained with a FN0205 mix (ATOMET 1001 admixed with 2.5 wt% Ni and 0.6 wt% Graphite) containing 0.6 wt% of a high melting point lubricant ensuring good flowability, good compressibility and low ejection forces in warm conditions. Mix was binder-treated with a patented blending technique [8-10] to produce a FLOMET mix with improved flowability and homogeneity. Proper adjustment was made to the binder treatment to produce a mix suitable for warm compaction.

Green and sintered properties were determined on standard TRS specimens pressed on a hydraulic lab press at room temperature and 150°C. Compacting pressure was 50 tsi (690MPa). Sintering was done at 1120 °C for 25 min in a $90\%N_2/10\%H_2$ atmosphere. Springback and dimensional change (D.C.) from die size were measured. Flow and apparent density (A.D.) were determined using MPIF standard procedures.

### Warm Compaction Test

Warm compaction tests were carried out on a 220 ton Cincinnati mechanical press model # 220-DCII-6 equipped with powder and die heating system facilities [11]. A die with an outside diameter of 2.166 in. (5.502 cm) and a core rod of 1.484 in. (3.769 cm) was used to press ring specimens. The effects of compacting pressure, powder temperature and die fill were investigated. Die, punches and core rod were heated at a temperature of 121°C for warm compaction trials and parts were pressed at a rate of 10 parts/min. The compaction conditions (part number, crown tonnage, powder temperature, die fill, strokes per minute, etc...) during all the tests were recorded with a portable computer and transferred to a spreadsheet for analysis.

Several parts were collected for determination of weight, green density and springback. Green density was evaluated by the water displacement technique and by measuring the physical dimensions of rings. Excellent agreement between both techniques was found, with differences not exceeding 0.01 $g/cm^3$. Sintered density and dimensional change of several parts pressed in different conditions were also determined. Sintering conditions were identical to those used for TRS specimens pressed in the lab. Springback and dimensional change were determined relative to the outside diameter.

## LABORATORY CHARACTERISTICS

Table 1 summarizes the physical, green and sintered characteristics of a FN0205 mix containing 0.6% lubricant. Green densities of 7.18 g/cm$^3$ and 7.30 g/cm$^3$ were respectively reached at room temperature and 300°F (150°C), representing 96.4 and 98.0% of the pore free density. Sintered densities of 7.25 and 7.32 g/cm$^3$ were reached on specimens pressed at room temperature and 300°F. Springback of specimens pressed in those conditions was 0.27% and 0.22% while dimensional change from die size was slightly negative at room temperature and slightly positive at 150°C.

Table 1: Physical, Green and Sintered Properties of the FN0205 FLOMET Mix as Determined in Laboratory.

| Flow Hall, s/50g | A.D. g/cm$^3$ | Comp. Temperature °F | Green Density g/cm$^3$ | Green Strength psi | Springback % | % Pore Free Density | Sintered Density g/cm$^3$ | T.R.S. kpsi | D.C. % | Hardness HRB |
|---|---|---|---|---|---|---|---|---|---|---|
| 27 | 3.08 | 22 | 7.18 | 1875 | 0.27 | 96.4 | 7.25 | 173 | -0.04 | 81 |
| | | 150 | 7.30 | 3800 | 0.24 | 98.0 | 7.32 | 183 | +0.06 | 81 |

## EFFECT OF PROCESSING PARAMETERS ON DENSITY

### Green Density

Table 2 summarizes green and sintered properties of the FN0205 mix as obtained on a production press in different compaction conditions. Maximum green density of 7.31 g/cm$^3$, 7.26 g/cm$^3$ and 7.24 g/cm$^3$ was respectively reached at 50 tsi (690 MPa) with a die fill of 0.4, 1.0 and 1.5 in. (1.0, 2.5 and 3.8 cm) by warm compaction. This represents 98.0, 97.3 and 97.1% of the pore free density. Green density reached with a die fill of 0.4 in. (1.0 cm) is on a par with that obtained on a lab scale while it was 0.04 and 0.06 g/cm$^3$ lower with a die fill of 1.0 and 1.5 in. (2.5 and 3.8 cm) respectively. It should be noted that a part height very close to that of specimens pressed on a lab press was reached with a die fill of 0.4 in. (1.0 cm) while part height was twice with a die fill of 1.0 in. (2.5 cm) and three times higher with a die fill of 1.5 in. (3.8 cm) compared to lab specimens.

Figure 1 shows the effect of the powder temperature on green density at 30, 40, 50 and 55 tsi (415, 550, 690 and 760 MPa) for a die fill of 1.0 in. (3.8 cm). It can be observed for relatively low compacting pressures such as 30 and 40 tsi (415 and 550 MPa) that the green density increases continuously as the powder temperature is increased from room to above 150°C. The gain in density in these conditions is about 0.14 g/cm$^3$ versus cold compaction. However, at higher compacting pressures such as 50 and 55 tsi (690 and 760 MPa), green density reaches a maximum at about 230°F (110°C) which is maintained up to about 125°C and decreases above that. The reduction in green density past 125°C is larger at 55 tsi (760 MPa) than at 50 tsi (690 MPa). Gain in density obtained at 50 and 55 tsi (690 and 760 MPa) is 0.09 and 0.07 g/cm$^3$ respectively compared to cold compaction.

As illustrated in Figure 2, at a compacting pressure of 50 tsi (690 MPa), increasing the die fill from 0.4 in. (1.0 cm) to 1.5 in. (3.8 cm) leads to a reduction in green density at any temperatures, except at room temperature where density at 1.5 in. (3.8 cm) is slightly higher than that at 1.0 in. (2.5 cm). Green density increases continuously as the powder temperature is increased for the lower die fill tested while it first increases, reaches a maximum and then decreases for the two other die fills. The powder temperature which maximizes green density is found to decrease when die fill is increased. Maximum green density is reached at 150°C for a die fill of 0.4 in. (1.0 cm), and 110°C for a die fill of 1.0 in. (2.5 cm) and 93°C for a die fill of 1.5 in. (3.8 cm).

It is clear from these results that the optimum powder temperature to maximize green density is a function of the compacting pressure and the die fill. This is an important point to consider when prototyping a new part.

Table 2: Summary of Green and Sintered Properties of a FN0205 Mix Processed on a Production Press.

| Compaction Conditions | | | Green Density | Springback | Sintered Density | D.C., |
|---|---|---|---|---|---|---|
| Die Fill, in. (cm) | Powder Temp, °C | Comp Pres, tsi (MPa) | g/cm$^3$ | % | g/cm$^3$ | % |
| 0.4 (1.0) | Room | 50.6 (697) | 7.21 | 0.16 | 7.23 | -0.12 |
| | 88 | 49.8 (686) | 7.28 | 0.15 | 7.29 | -0.07 |
| | 110 | 51.0 (703) | 7.31 | 0.16 | 7.32 | -0.07 |
| | 126 | 51.3 (707) | 7.31 | 0.16 | 7.32 | -0.09 |
| | 145 | 50.6 (697) | 7.31 | 0.16 | 7.32 | -0.10 |
| 1.0 (2.5) | Room | 39.7 (547) | 7.01 | 0.19 | - | - |
| | | 51.2 (706) | 7.16 | 0.23 | 7.25 | -0.16 |
| | | 55.2 (761) | 7.21 | 0.22 | - | - |
| | 90 | 39.8 (549) | 7.10 | 0.18 | - | - |
| | | 50.6 (697) | 7.25 | 0.23 | 7.30 | -0.11 |
| | | 56.0 (772) | 7.28 | 0.23 | - | - |
| | 110 | 40.6 (560) | 7.13 | 0.16 | - | - |
| | | 49.6 (684) | 7.26 | 0.22 | 7.31 | -0.12 |
| | | 55.7 (768) | 7.29 | 0.26 | - | - |
| | 125 | 40.2 (554) | 7.15 | 0.16 | - | - |
| | | 51.1 (704) | 7.26 | 0.26 | 7.34 | -0.13 |
| | | 55.1 (704) | 7.28 | 0.32 | - | - |
| | 142 | 39.8 (549) | 7.16 | 0.16 | 7.20 | -0.18 |
| | | 50.0 (689) | 7.24 | 0.32 | 7.35 | -0.14 |
| | | 55.4 (764) | 7.25 | 0.43 | 7.38 | -0.10 |
| 1.5 (3.8) | Room | 49.9 (688) | 7.19 | 0.21 | 7.24 | -0.14 |
| | 95 | 50.7 (699) | 7.24 | 0.32 | 7.30 | -0.05 |
| | 112 | 50.8 (700) | 7.24 | 0.36 | 7.32 | -0.07 |
| | 127 | 51.5 (710) | 7.23 | 0.46 | 7.34 | -0.08 |
| | 142 | 50.3 (693) | 7.21 | 0.49 | 7.34 | -0.09 |

(1) Die Temp: 121°C, Strokes per min.: 10 parts/min.

**Sintered Density**

Sintered properties of rings pressed under different conditions at 50 tsi (690 MPa) are summarized in Table 2. Sintered densities reached between 110 and 150°C were generally equal or slightly higher than that obtained in the lab at 150°C (7.32 g/cm$^3$). In fact, sintered densities up to 7.35 g/cm$^3$ at 50 tsi (690 MPa) and 7.37 g/cm$^3$ at 55 tsi (760 MPa) were reached by warm compaction. It should be noted that such sintered densities were obtained even if green density was significantly lower than that obtained in the lab.

Figure 3 shows the effect of the compacting pressure on the green and sintered density of parts pressed at a powder temperature of around 145°C. It could be observed that sintered density increased continuously with the compacting pressure contrary to the green density, which reached a maximum at 50 tsi (690 MPa) and remained stable over that. Gain in density after sintering is found to increase as the compacting pressure increased.

Figure 4 shows the variation of the green and sintered density as a function of the powder temperature at 50 tsi (690 MPa) for different die fills. It was found that increasing the powder temperature from room to around 150°C led to an increase in sintered density for any die fill even if reduction in green density was observed for a die fill of 1.0 and 1.5 in. (2.5 and 3.8 cm). In fact, very similar sintered densities were obtained with all die fill even if large differences in green density were found. Sintered density is even slightly higher for parts pressed with the largest die fills. The larger gain from green to sintered density as the die fill increases indicates that larger amount of energy is stored in larger part during compaction.

**Relationship Between Density and Springback**

Green density is a function of the compressibility characteristics of the powder which can be measured by the density reached in the die during compaction and the volume expansion undergone by parts during ejection known as springback. Figure 5 shows the effect of powder temperature on the density reached in the die for a die fill of 1.0 in. (2.5 cm). It was calculated by using the die surface area perpendicular to the compaction axis and the part green height. Densities slightly higher than those calculated were reached considering that the height likely underwent an expansion at ejection. However, amplitude of that expansion is unknown. It can be observed that the density reached in the die increases as the powder temperature is increased to reach about 7.33 g/cm$^3$ at 150°C. Similar results were obtained with other die fills. It is clear that the reduction in green density when temperature past a given point as observed in different conditions and as illustrated in Figure 5 is not due to a loss in compressibility which was in fact improved by the powder temperature during compaction but rather to a larger springback. Relation between the springback and the reduction in density at ejection which is the difference between density reached in the die and green density is shown in Figure 6 for parts pressed under different conditions.

Figure 7 shows the springback as a function of powder temperature for different compacting pressures and a die fill of 1.0 in. (2.5 cm) It can be seen that the compacting pressure has a strong effect on the springback as the powder temperature increases. For low compacting pressures such as 30 and 40 tsi (415 and 550 MPa), the powder temperature is found to have almost no effect on the springback which decreases slightly at 30 tsi (415 MPa) and remains relatively stable at 40 tsi (550 MPa) as the temperature is increased. This explains why green density in Figure 1 was found to increase continuously as the powder temperature increased for such compacting pressures. However, for higher compacting pressures such as 50 tsi (690 MPa) and 55 tsi (760 MPa), the powder temperature has a strong influence on the springback which increases significantly when temperature is increased over a given point (about 120°C at 50 tsi and 95°C at 55 tsi). Increasing the compacting pressure is found to enhance the effect of the temperature on the springback by decreasing in particular the temperature at which the springback starts to increase.

Figure 8 illustrates the relation between the springback and the powder temperature for different die fills at a compacting pressure of 50 tsi (690 MPa). Increasing the die fill from 0.4 in. to 1.5 in. (1.0 to 3.8 cm) increases the springback at any temperature, which explains the reduction in green density observed when die fill is increased. In fact, compared to the intermediate die fill (discussed previously), decreasing the die fill to 0.4 in. (1.0 cm) has suppressed the effect of the powder temperature on springback which remained stable for the entire temperature range while increasing the die fill to 1.5 in. (3.8 cm) has the opposite trend. The variation of springback with powder temperature for different die fills shown in Figure 8 explains why green density increases continuously with the very low die fill while it decreases beyond an optimum temperature for higher die fills.

It was shown in Figures 3 and 4 that increasing the powder temperature was beneficial to the sintered density while die fill has no real effect. Figure 9 shows the effect of springback on the gain in density after sintering for rings pressed under different conditions. It is clear that the gain in density is a direct function of springback. This suggests that springback, which is known as an elastic expansion when compacting pressure is removed and part is ejected from die, is also a direct measure of the energy stored in parts during compaction, which acts as a driving force for diffusion and densification during sintering.

## PROCESS ROBUSTNESS AND CAPABILITY OF MIXES

Figure 10 shows the effect of powder temperature on the apparent density for binder treated and regular FN0205 mixes. Apparent density was measured by the ratio of the weight to volume in the die prior to compaction. It could be observed that the binder treated mix shows a much more stable apparent density between 80 and 145°C than the regular mix. This results in a much better weight stability during warm compaction for the binder treated mix, especially if some variations in the powder temperature occurred during processing. In addition, binder was also found to help stabilize the powder temperature by homogenizing and regulating the heat absorption capacity of powder because the thermal conductivity is reduced. According to Figure 10, very good results in terms of powder stability to a temperature change is obtained when the powder temperature is set between 80 and 145°C), which corresponds to the optimum compacting temperature for green density.

Production run trials of 300 to 425 parts were carried out with FLOMET FN0205 mixes containing either 0.6 or 0.5% lubricant to evaluate the process capability and stability in terms of part to part weight, density, compacting pressure and temperature variations. Runs were done at different powder temperatures and at a compacting pressure of around 50 tsi (690 MPa), a production rate of 10 parts/min and a die fill of 1.0 in. (2.5 cm). Tooling temperature was set at 121°C. Five parts out of every 25 were collected for weight and density evaluation.

Table 3 summarizes the results obtained for five runs and Figure 11 shows the process charts for two of these runs. The powder temperature varied within "0.8 to " 2.5°C in all cases. However, it can be observed in Figure 11 that the temperature was maintained within "0.6°C when stabilization was reached. Data and parts were collected even if stabilization was not totally reached to evaluate the materials robustness to a temperature variation. Runs with the mix containing 0.6% lubricant gave green density of 7.25, 7.25 and 7.24 g/cm$^3$ at 83, 124 and 145°C respectively while a green density of 7.29 g/cm$^3$ was reached with the 0.5% lubricant mix at either 124 or 145°F. Sintered densities between 7.30 to 7.33 g/cm$^3$ and 7.35 to 7.37 g/cm$^3$ were respectively reached for the 0.6% and 0.5% lubricant mixes. As mentioned previously, the sintered density increases as the processing powder temperature is increased. Ranges (R) for the compacting pressure and the part weight were 1.5 to 2.3 tsi (21 to 32 MPa) and 0.98 to 1.64 g respectively for four of these runs and 2.8 tsi (39 MPa) and 2.02 g for one run performed at 145°C with the FN0205-0.6% lubricant mix. Ranges obtained for the part weight represent a variation of " 0.41 to " 0.84% which shows the excellent behaviour and capability of FLOMET mixes specifically engineered for warm compaction. Variation in compacting pressure resulted in a variation of " 0.005 to " 0.015 g/cm$^3$ in green density and similar variation for the sintered density.

Table 3 Summary of Production Runs Carried out with FLOMET FN0205 Mixes Containing 0.5 or 0.6% Lubricant (1).

| Lub content % | Powder Temp. °C | | Comp. Pres. tsi (MPa) | | Part Weight g | | | Green Density g/cm$^3$ | | Sintered Density g/cm$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|
| | Avg | R | Avg | R | Avg | R | %Variation | Avg | R | |
| 0.5 | 123 | 5.0 | 50.3 (693) | 1.6 (22) | 120.4 | 1.3 | 0.55 | 7.29 | 0.01 | 7.35 |
| | 143 | 3.3 | 50.3 (693) | 1.8 (25) | 120.9 | 1.4 | 0.56 | 7.29 | 0.02 | 7.37 |
| 0.6 | 83 | 1.7 | 49.6 (684) | 2.3 (32) | 120.4 | 1.6 | 0.66 | 7.26 | 0.03 | 7.29 |
| | 124 | 2.8 | 49.6 (684) | 1.5 (21) | 120.6 | 1.0 | 0.41 | 7.25 | 0.02 | 7.33 |
| | 145 | 2.8 | 50.1 (691) | 2.7 (37) | 120.6 | 2.0 | 0.84 | 7.24 | 0.01 | 7.34 |

(1) Die Temp: 121°C, Die fill: 1.0 in. (2.5 cm), strokes per min.: 10 parts/min.

## CONCLUSIONS

Tests were conducted on a production press equipped with warm compaction capability to study the effect of processing parameters such as compacting pressure, powder temperature and part height on the green and sintered properties of binder treated FN0205 materials. The following conclusions can be drawn from this work:

1) There is an optimum powder temperature range to maximize green density on a production press. The optimum temperature range to reach maximum green density was found to be a function of the compacting pressure and the part height. The larger the compacting pressure and/or the die fill, the lower the optimum powder temperature. Optimum powder temperature varies from 150°C to around 93-110°C for part heights varying from 0.21 in. (0.5 cm) to 0.75 in. (1.9 cm).

2) Sintered density increases with temperature from room temperature to 160°C under any compacting condition. Sintered densities varying from 7.30 to 7.34 g/cm$^3$ were reached between 93 and 150°C at 50 tsi (690 MPa).

3) Increasing the part height has no deleterious effect on the sintered density which instead increases slightly with the die fill.

4) Reduction in green density as temperature exceeds an optimum point for compacting pressures above 40 tsi (550 MPa) is related to an increase of springback at ejection. Optimum temperature is that which maximizes compressibility and minimizes springback.

5) The gain in density during sintering is a direct function of the springback. The higher the springback, the higher the density gain. A good sintering ensures complete recovery of density loss due to springback at ejection.

6) Binder treatment improves flowability, stabilizes the apparent density between 82 and 145°C and allows a much better temperature control by regulating the heat transfer to powder. This results in excellent weight stability during warm compaction for FLOMET mixes.

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