Presented at the 2010 MPIF / APMI International Conference on Powder Metallurgy & Particulate Materials held in Ft Lauderdale, USA in 2010 and published in the Advances in Powder Metallurgy & Particulate Materials - 2010 conference proceedings available from the Metal Powder Industries Federation.

Effect of powder characteristics, mix formulation and compacting parameters on green density of PM parts

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ABSTRACT

High density PM parts can be produced to densities exceeding 7.5 g/cm³ but generally require additional processing steps that negatively affect their costs. Many R&D projects are currently carried out on both the materials and compaction techniques to reach high density at an affordable cost. The objective of this paper is to review how the powder characteristics, the additives in the mix formulation (specifically lubricant and graphite) and the compaction parameters affect densification during compaction. Results showed that at compacting pressures below 620 MPa (45 tsi), powder compressibility is a key parameter to achieve high density, while above 690 MPa (50 tsi), the low density additives (lubricant and graphite) have the largest impact. New polymeric lubricants admixed in lower concentrations can be used to maximize green density but the most important variable is still the application of high compacting pressures.

INTRODUCTION

The growth of the PM market relies on the development of new technologies to produce PM parts that will fulfill demanding requirements in highly loaded applications. In order to meet such requirements PM parts must be manufactured to high density at a competitive cost. A good understanding of the powder behaviour during compaction is also required to determine the maximum achievable green density that can be reached for specific steel powder grades, mix formulations and compaction processes. Densification during compaction occurs generally in three steps [1,2]. The first step is characterized by a rapid rate of densification as the pressure is applied, corresponding to particle rearrangement. The second stage is characterized by a reduction of the densification rate as the level of porosity decreases, which leads to the formation of new particle contacts and to plastic deformation of particles. These plastic deformations cause work hardening which makes the densification more difficult as the pressure increases. Materials exhibiting high yield strengths would show a significant reduction in the rate of densification during this step of compaction compared to those with low yield strengths. In addition to the softness of the particles, their shape and particle size distribution will also have an effect on the densification during compaction. Finally, the third compaction step is characterized by a very low rate of densification due to the high level of work hardening of the particles.

The green density achieved during compaction is also dependant on the mix formulations. Addition of low density additives such as lubricants and graphite will significantly affect the maximum achievable green density. At low compacting pressure, addition of a larger concentration of lubricant promotes densification as the pressure is increased because it facilitates particle rearrangement. However, at high pressure, as the lubricant is located in the residual porosity, it impedes densification and limits the maximum achievable green density [3,4,5,6,7]. The development of new lubricating systems has made possible the reduction in their concentrations in the powder mixes and hence increases the maximum achievable green density, [3,7].

The third aspect to take into account is the compaction process itself. Powder forging and double pressing double sintering are known processes that make it possible to reach density above 7.4 g/cm³ but their higher cost limits their use to niche markets. Other technologies such as high velocity compaction (HVC), dynamic magnetic compaction (DMC) and high pressure combustion driven powder compaction (CDC) have been evaluated but again are limited to specific simple part shapes [8,9,10,11,12,13,14]. Another route to produce high density parts is to raise both the powder and die temperature in a temperature range of 80 to 175°C. Many studies have been published on this technology that makes it possible to gain between 0.1 to 0.3 g/cm³ in green density compared to conventional compaction [15,16,17,18,19]. This process requires that both the powder and the die components have to be heated within a relatively tight range of temperatures which limits the growth of this technology. When combined with die wall technology and warm compaction at high compacting pressures, up to 1960 MPa, green density in the order of 7.7 g/cm³ has been reported [20]. In recent years, new lubricating systems have been developed to operate at lower temperatures and by heating only the die up to about 95°C, small parts can be pressed up to 7.4 g/cm³ [3,15,21,22, 23]. It is worth noting that all these compaction technologies can be combined with a surface densification operation after sintering to further increase the static and dynamic properties.

The objective of this paper is to review the effect of the various variables involved during compaction on the maximum achievable green density in order to better align future R&D studies.

EXPERIMENTAL PROCEDURE

The two powder grades used in this study were ATOMET 1001HP, powder A, and ATOMET 4401, powder B. Table 1 shows the typical chemical and physical properties of these powders. Powder A is a pure iron powder exhibiting a high compressibility while powder B is a 0.85% Mo low alloy steel powder having a good compressibility.

Table 1. Chemical and physical properties of powders A and B.

Powder	C, %	O, %	S, %	Mn, %	Mo, %	+100 mesh, %	-100/+325 mesh, %	-325 mesh, %
A	0.004	0.05	0.004	0.038	0.001	19.2	66.0	14.8
B	0.004	0.10	0.007	0.16	0.82	10.2	65.7	24.1

In a first part of the study, mixes were prepared with 2% Ni (Inco T110D) and 0.35% graphite and either 0, 0.15, 0.25, 0.50 or 0.75% EBS wax and pressed at either 550, 690, 830, and 950 MPa with the die heated to 50°C. For mixes with less than 0.50% lubricant, the die walls were lubricated with zinc stearate.

In a second part of the study, mixes with 2% Ni and 0.35% graphite were prepared with both powders and either 0.3 or 0.45% of a proprietary HD lubricant and pressed at 75°C to evaluate the effect of this experimental lubricant on green density. All the compaction trials were carried out with a hydraulic press and a tool steel die. Finally, some specimens from mixes based on powder B were sintered at 1205°C for 45 minutes in a 90% nitrogen and 10% hydrogen atmosphere to evaluate the evolution of the density from the green to sinter state.

RESULTS AND DISCUSSION

Tables 2 and 3 summarize the green density and springback values reached for each mix formulation and compacting pressure.

Table 2. Green density and springback values reached for the different mixes and compacting pressures for powder A.

Pressure	0.75% EBS		0.5% EBS		0.25% EBS		0.15% EBS		0% EBS	
MPa	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %
550	7.157	0.272	7.163	0.264	7.184	0.264	7.189	0.264	7.150	0.240
690	7.247	0.304	7.299	0.304	7.339	0.304	7.357	0.296	7.358	0.288
830	7.303	0.312	7.388	0.312	7.440	0.328	7.459	0.320	7.487	0.296
950	7.309	0.328	7.411	0.352	7.480	0.344	7.513	0.336	7.553	0.328

G.D.: green density
S.B.: springback

Table 3. Green density and springback values reached for the different mixes and compacting pressures for powder B.

Pressure	0.75% EBS		0.5% EBS		0.25% EBS		0.15% EBS		0% EBS	
MPa	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %	G.D., g/cm³	S.B. %
550	7.061	0.264	7.043	0.248	7.089	0.264	7.042	0.256	7.073	0.248
690	7.199	0.288	7.236	0.288	7.258	0.312	7.282	0.304	7.275	0.288
830	7.270	0.328	7.336	0.328	7.372	0.336	7.407	0.328	7.420	0.312
950	7.299	0.352	7.385	0.344	7.441	0.360	7.460	0.344	7.500	0.312

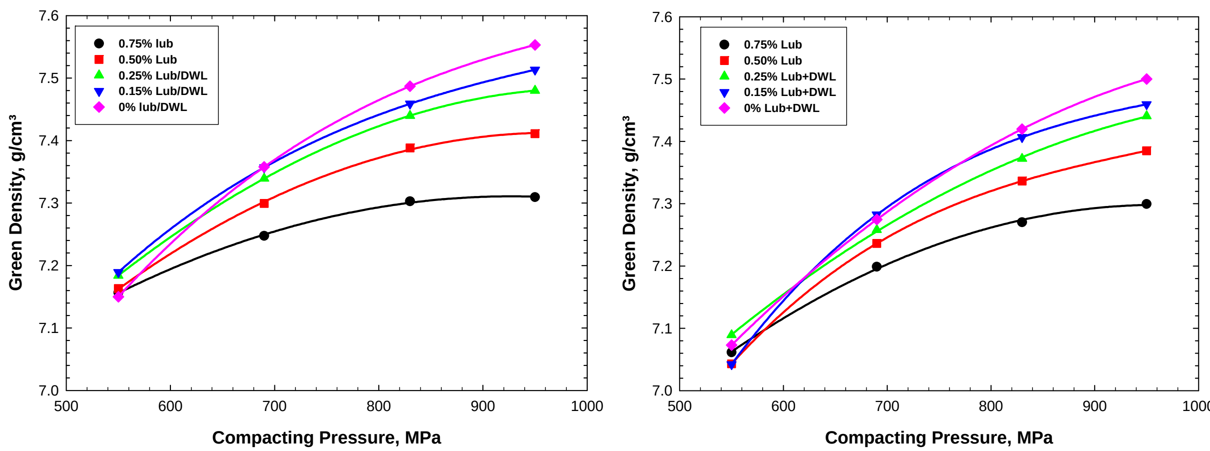
G.D.: green density
S.B.: springback

The compressibility curves for powders A and B with the various concentrations of lubricant are illustrated in Figure 1. Both powders show similar compaction behaviour with however higher green density values reached with specimens pressed with mixes based on powder A. At the lowest compacting pressure, 550MPa, the highest green densities are reached at 0.15 and 0.25% lubricant for powder A and at 0.25% lubricant for powder B. This confirms the beneficial effect of lubricant during the first stage of compaction, i.e. when the particle rearrangement occurs. On the other hand, at 950 MPa, the highest density values are reached with the materials without lubricant and the lowest one with the materials containing 0.75% lubricant, confirming the detrimental effect of lubricant when approaching the pore free density limits.

This is better illustrated in Figure 2, where the variation of the density with compacting pressure for the various levels of lubricant is expressed as a function of the relative density. Now, the powder mixes exhibiting the highest relative green density values are those containing 0.75% lubricant and the lowest ones with the mixes without lubricant. It is worth noting that the relative density levels off at about 98% of the pore free density. The major cause of this behaviour can be related to the springback after ejection, which induces an increase of the volume of the specimens for the same mass, leading to a loss in density. As illustrated in Figure 3, the springback increases linearly with the compacting pressure, with lower values measured for the specimens pressed from the lubricant free mixes. It is worth noting that both powders show similar springback values for a specific compacting pressure. Therefore, as the relative density approaches 98%, raising the compacting pressure to increase the density increases the springback and hence results in an only marginal gain in density.

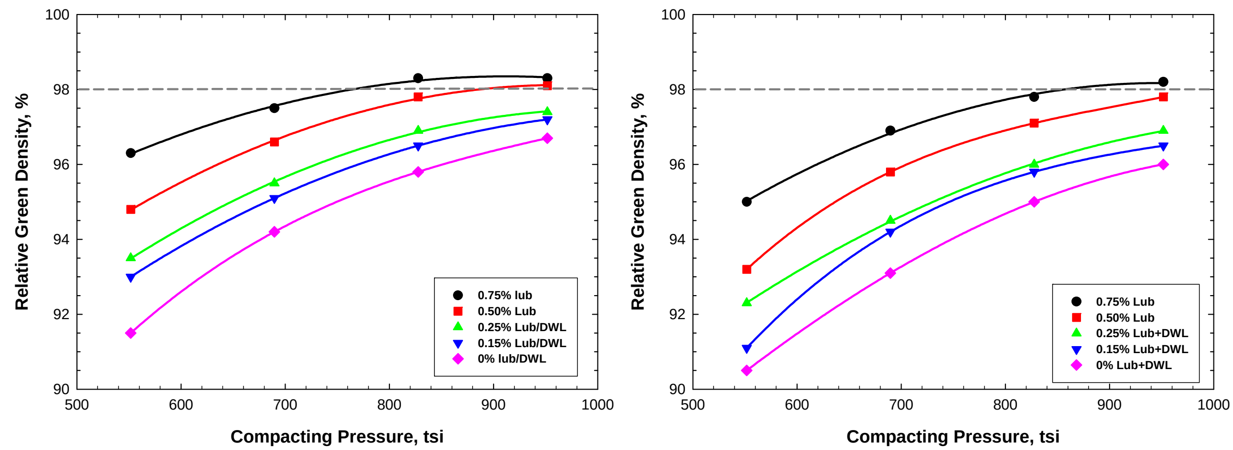
Another interesting conclusion that can be drawn from the compressibility curves illustrated in Figure 2 is that at low compacting pressure, 550 MPa, the powder compressibility plays an important role in the maximum achievable green density at that given pressure and the effect of lubricant concentration is marginal. This is better illustrated in Figure 4, which shows the effect of lubricant concentration on green density reached with specimens pressed with powder A and powder B, pressed at either 550 or 950 MPA. Specimens pressed at 550 MPa from mixes based on powder A achieved, on average, 7.17 g/cm³ while those pressed with mixes containing powder B, achieved 7.06 g/cm³. For both powders, at that compacting pressure, the effect of lubricant concentration is negligible.

In contrast, at 950 MPa, the lubricant level is now the major contributor to green density, particularly at 0.75% lubricant. Indeed, at 0.75%, both powders show similar green density values while at 0% lubricant, the difference between both powders is only 0.05 g/cm³, compared to 0.11 g/cm³ at 550 MPa.



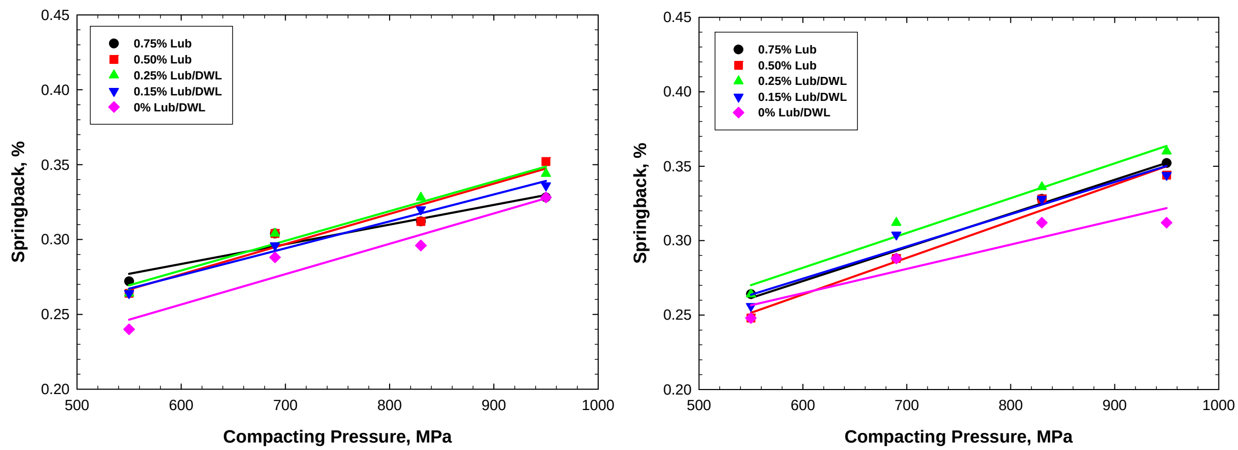
POWDER A **POWDER B**

Figure 1. Variation of green density with compacting pressure and lubricant concentration for powders A and B (mixes with 035% graphite, 2% Ni and x% lubricant).



POWDER A **POWDER B**

Figure 2. Variation of the relative green density with compacting pressure and lubricant concentration for powders A and B (mixes with 035% graphite, 2% Ni and x% lubricant).



POWDER A **POWDER B**

Figure 3. Variation of the springback of the specimens pressed with powders A and B with compacting pressure and lubricant concentration (mixes with 0.35% graphite, 2% Ni and x% lub).

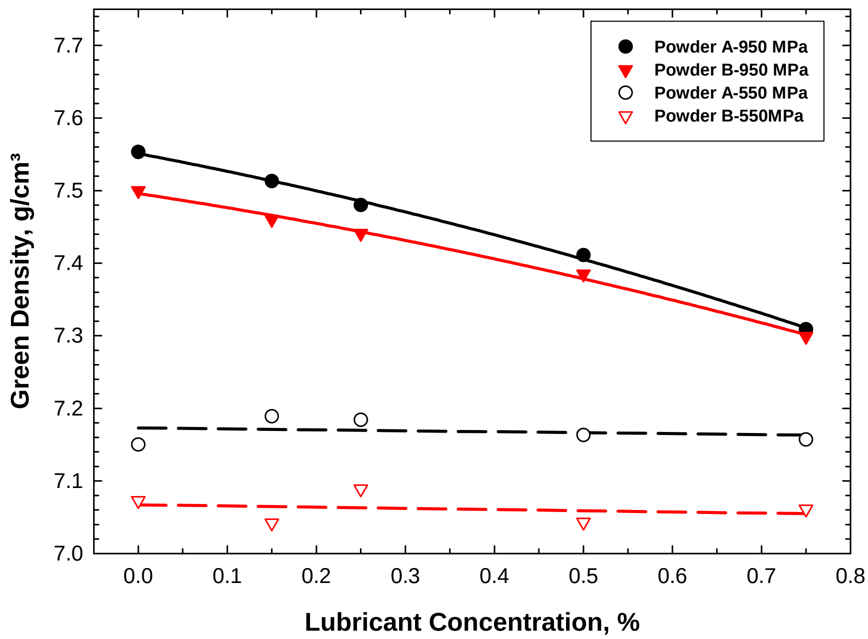


Figure 4. Effect of lubricant concentration on green density of specimens pressed with powders A and B at 550 and 950 MPa (mixes with 0.35% graphite, 2% Ni).

Similar to lubricants, graphite is also a low density additive which, when admixed to steel powders limits the maximum achievable green density. However, its effect is less detrimental than lubricant. As illustrated in Figure 5, which shows the theoretical variation of green density with lubricant and graphite concentrations for powder B, a variation of the lubricant concentration of 0.1% induces a change of 0.05 g/cm^3 of the green density while the same variation for the graphite concentration results in a change of only 0.02 g/cm^3. For a concentration of graphite of 0.35% and 2% Ni, at 0.4% lubricant, the theoretical maximum green density that could be reached is 7.59 g/cm^3. However, based on the 98% relative density limit, the maximum value would be reduced to about 7.44 g/cm^3, a value close to the one that can be interpolated in Figure 4.

The development of new lubricant systems with improved lubrication makes it possible to increase the green density by lowering the lubricant concentration. However, these new lubricants need to be used in a temperature range of 60 to 80°C to favour lubricant movement toward the die walls as the pressure increases [23]. This lubricant behaviour leads to an improvement of the lubrication at the die walls during ejection and a reduction of the lubricant inside the part, making it possible to achieve a higher green density. Also, using a warm die at about 80°C contributes to significantly decrease the yield strength of iron particles [24], leading to an increase of the densification during compaction. As illustrated in Figure 6, yield strength of pure iron decreases by about 40 MPa when the temperature is raised from room temperature to 80°C. A further increase up to 150°C, temperature often cited for warm compaction where both the powder and the die components have to be heated, results in a decrease of only 26 MPa. Therefore, 60% of the reduction of the yield is reached between 25 and 80°C and only 40% between 80 and 150°C. Above 150°C, the reduction in the yield values is marginal.

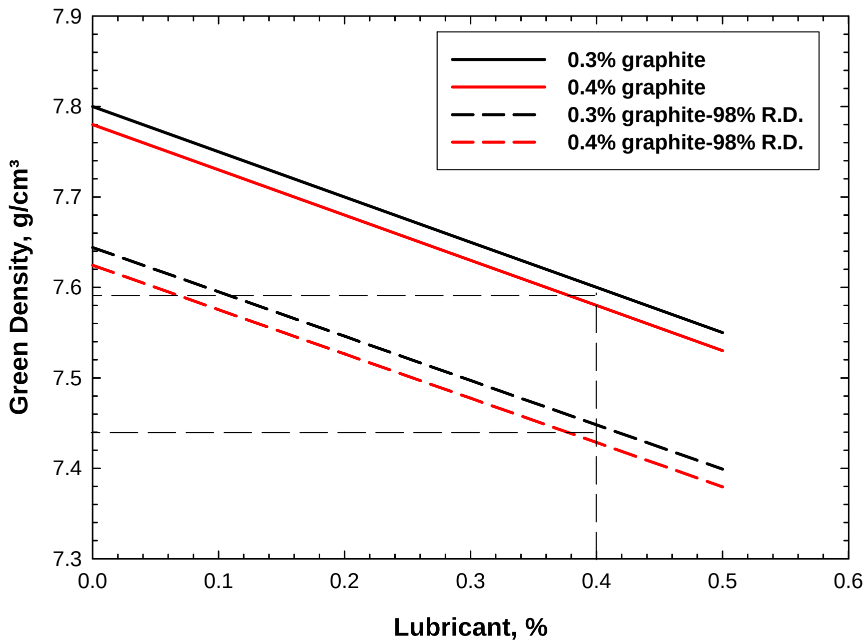


Figure 5. Effect of the concentration in lubricant and graphite on green density at for levels of 100 and 98% relative green density (mixes made from powder B with 2% Ni).

Table 4 summarises the green density and springback values measured when compacting specimens with powder B with 0.35% graphite, 2% Ni and either 0.3 or 0.4% HD lubricant. Figure 7 illustrates the variation of green density with compacting pressure for the mix based on powder B with 0.35% graphite, 2% Ni and either 0.3 or 0.4% HD lubricant. The compressibility curves of the mix without lubricant and with 0.15% wax pressed with the die walls lubricated, Figure 1, are also illustrated for reference purpose. At 550 MPa, the specimens pressed with the HD lubricant show significantly higher green density values than those of the references, even higher than those reached with the mix containing 0.75% wax (Figure 1). Therefore, even if the amount of lubricant is decreased, it significantly improves particle rearrangement at the initial stage of compaction. It is also worth noting that the specimens pressed with the mix containing 0.4% HD lub achieves higher green density values than those pressed with the mix with 0.3% HD lub at 550 and 690 MPa. However, the green density levels off at about 7.47 g/cm³ when the compacting pressure reached 950 MPa. It is worth noting that the value reached at this compacting pressure is comparable to that reached with the mix containing 0.15% wax pressed at 50°C with the die walls lubricated. In contrast, for the specimens pressed with the mix containing 0.3% HD lub, the green density is still increasing to reach 7.51 g/cm³ at 950 MPa, a value similar to that reached with the mix without lubricant pressed with the die walls lubricated.

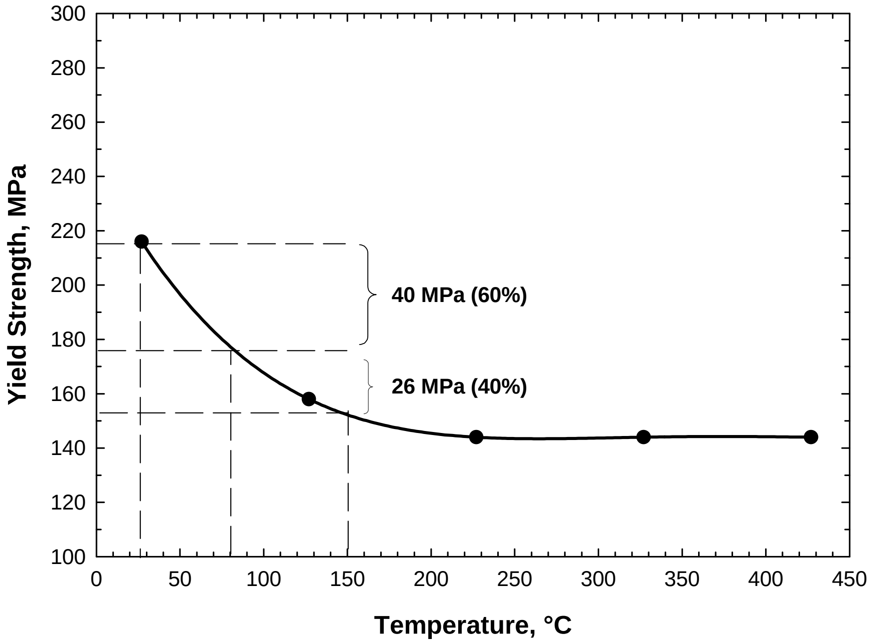


Figure 6. Calculated yield strength of pure iron with temperature [24].

Table 4. Green densities and springback values reached for the mixes based on powder B with 0.35% graphite, 2% Ni and either 0.3 or 0.4% HD lubricant for the different compacting pressures for powder B.

Pressure	0.3% HD		0.4% HD	
MPa	Green density, g/cm³	Springback, %	Green density, g/cm³	Springback, %
550	7.135	0.280	7.149	0.280
690	7.327	0.320	7.335	0.320
830	7.438	0.352	7.447	0.384
950	7.512	0.392	7.468	0.408

Figure 8 illustrates the variation of the springback values with compacting pressure for specimens pressed with the mix based on powder B with either 0.3 or 0.4% HD lubricant. The values measured without and with 0.15% wax and die wall lubrication, Figure 3, are also given for reference purpose. The springback values increases with the compacting pressure, with a steeper slope at 0.4% HD lub. It is also worth noting that the springback values with the HD system are higher than those measured with the mixes without lubricant and with 0.15% wax pressed with a lubrication of the die walls.

A final consideration to maximize the density of PM materials is the selection of a mix formulation that will show densification during sintering. This is the reason why formulations with nickel are usually preferred to promote shrinkage and the addition of copper is prohibited because it favours growth during sintering. Figure 9 compares the green and sintered densities measured at various compacting pressures for the mix based on powder B with 0.35% graphite, 2% Ni and either 0.3 or 0.4% HD lubricant. Below 830 MPa, the gain in density between the green and sintered states is, on average, similar for both lubricant concentrations with a value of about +0.05 g/cm³. On the other hand, above 830 MPa and more particularly at 950 MPa, the gain in density between both states increases to reach 0.08 g/cm³ at 950 MPa for the specimens pressed with the mix containing 0.4% lubricant. This indicates that part of the loss in the green density due to the larger springback at high compacting pressure is recovered during sintering

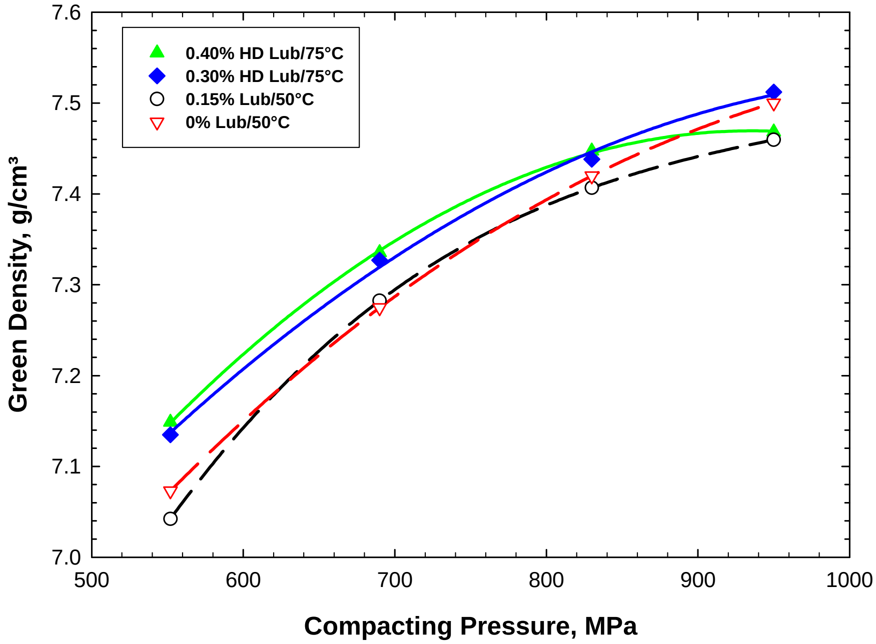


Figure 7. Variation of green density with compacting pressure and lubricant concentration (Powder B+0.35% graphite, 2% Ni and x% lubricant).

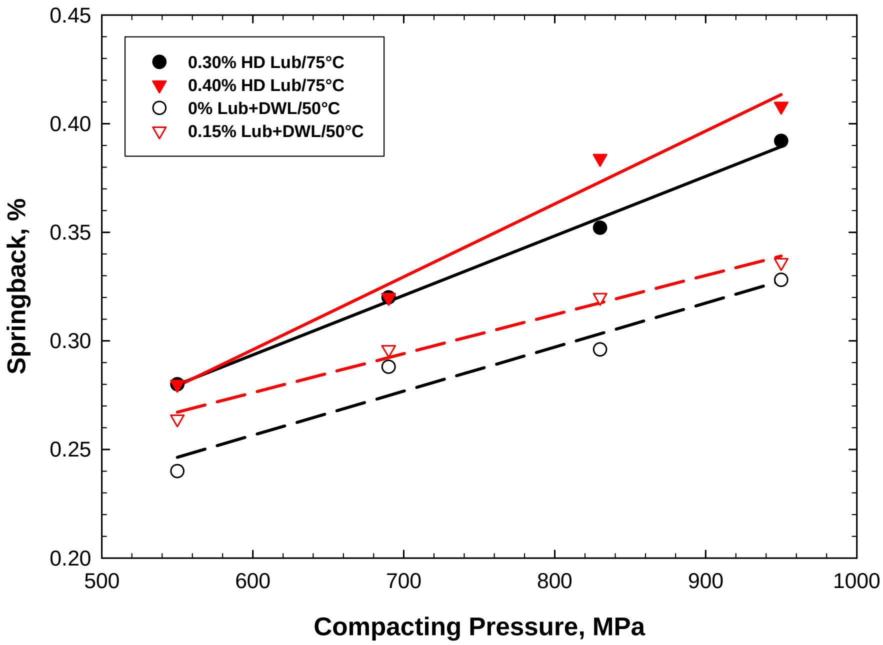


Figure 8. Variation of the springback of the specimens pressed with powder B with compacting pressure and lubricant concentration (mixes with 0.35% graphite, 2% Ni and x% lub).

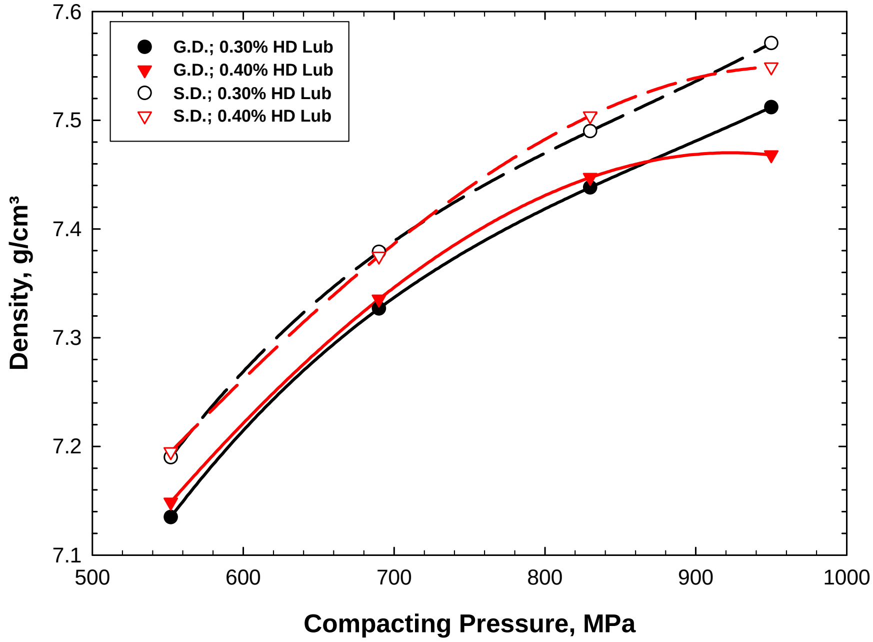


Figure 9. Variation of the green and sintered densities with compacting pressure (powder B with 0.35% graphite, 2% Ni and x% HD lubricant).

CONCLUSIONS

Based on the results reached in this study with powders A and B:

1. At low compacting pressure, 550 MPa, powder compressibility plays an important role in the maximum achievable green density while the effect of lubricant concentration is marginal.

2. At 950 MPa, the lubricant concentration is the most important contributor limiting the maximum achievable green density while powder compressibility plays a more marginal role.

3. Springback increases when the compacting pressure increases, which limits the maximum achievable green density to about 98% of the relative density.

4. The development of new polymeric lubricants with improved lubrication efficiency make it possible to reduce their concentrations in the powder mixes and hence, achieve higher green densities.

5. The gain in density between the green and sintered states increases with compacting pressure, indicating that a loss in density due to the larger springback values measured at high compacting pressures is recovered during sintering.

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