

STUDY OF COMPACTION AND EJECTION PROPERTIES OF POWDER MIXES PROCESSED BY WARM COMPACTION

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ABSTRACT

Warm compaction takes advantage of temperature to favor densification during compaction. However, this technique requires powder mixes with specific physical characteristics to be adequately processed in the temperature range involved in warm pressing. The mix formulation, especially the lubricant/binder system, should provide good flowability and compressibility and lubrication of the die walls to ensure high green densities and low ejection forces.

The behavior of powder mixes made with different lubricants processed by warm compaction was investigated with an instrumented compacting device to quantify the phenomena taking place during the compaction and ejection processes. In particular, the influence of compacting temperature and part height on the level of friction at the die walls, the intrinsic compactability, volume expansion and, the ejection stripping and sliding pressures are discussed.

INTRODUCTION

The warm compaction technique, which consists of pressing a preheated powder mix in a heated die [1], is a single press/single sinter process which allows an increase of green density, and, consequently, of sintered density [2,3]. Sintered densities ranging typically between 7.25 and 7.45 g/cm³ can be achieved by warm compaction at a compacting pressure not exceeding 690 MPa [3]. Even higher densities can be achieved by using slightly higher compacting pressure combined with high temperature sintering [4].

Warm compaction takes advantage of the fact that a moderate increase of the compacting temperature lowers the yield strength of steel particles and increases their ductility, leading to an increase of densification for a given applied pressure [5]. Typically, warm compaction is done at temperatures ranging between 100 and 150°C. In some cases, compaction can be done at higher temperatures up to 300°C. However, increasing the temperature above 150°C should have only a

moderate effect on green density based on works of Chagnon et al [3] and the curves of yield strength as a function of temperature for steel given in Bocchini's paper [6].

As it is the case for cold compaction, steel powder must contain lubricant specifically designed for warm compaction. The binder treatment of mixes also appears to be essential [7]. The lubricant/binder system should ensure a good and consistent feeding of the die and provide a low internal friction between particles and an excellent lubrication of die walls at the temperatures encountered during the compaction cycle in order to take advantage of the higher ductility of steel particles. The properties and intrinsic characteristics of lubricant and binder also determine the optimum compaction temperature.

Compressibility remains a key characteristic of powder mixes specifically designed for warm compaction. The compressibility of a powder mix is usually determined on laboratory presses using slugs or TRS specimens according to P/M standards. Most of the data on the compressibility of warm pressing mixes published so far in the literature were obtained with such test procedures. However, these procedures provide no information on the key phenomena taking place during compaction and ejection, i.e. the friction at die walls, the intrinsic compactability of powder and the volume expansion of compact at ejection. In addition, the size or aspect ratio of TRS specimens pressed at lab scale is much lower than that of parts commonly manufactured. The level of friction at die walls is much higher in the latter case. For these reasons, it is very difficult to use the results obtained on lab scale to predict the behavior of a mix under production conditions as discussed in a previous paper [8].

However, the use of a Powder Testing Center, an instrumented laboratory press, can be used to quantify the compaction and ejection processes and study the influence of compacting temperature on the characteristics of powder mixes. In particular, this press allowed quantifying of the three key properties or factors affecting the green density, namely the friction at die walls, the powder intrinsic compactability and the expansion at ejection. Different high melting point lubricants were evaluated in order to have a better understanding of their role and influence on the properties of warm pressed specimens. The properties of a new lubricant/binder system having an excellent lubricity at the temperatures typical of warm compaction are presented.

DENSIFICATION OF FERROUS POWDERS

The green density achieved after compaction in a closed die is a function of numerous factors. It depends typically on the compaction procedure and conditions, the tool materials, clearance and design, the part shape and complexity and the powder mix properties. However, for a given application and manufacturing conditions, the green density achieved is only a function of the mix characteristics. It is mainly affected by the apparent density and flow which determine the capacity of a powder to fill the die in a given time, the pore free density (which is the density if all the porosity is eliminated) and the compressibility.

The compressibility is the relation between the green density and the applied pressure. It is usually expressed by the applied pressure needed to reach a required green density or by the green density achieved at a given applied pressure [9]. However, the compressibility is dependent on three key phenomena, which take place during the compaction process:

- 1- The intrinsic ability of the powder to be densified in the absence of friction at die walls named compactability,
- 2- the friction between particles and die walls,
- 3- the expansion or the springback at ejection.

It should be emphasized that the compactability is only dependent on the intrinsic mechanical behavior of the powder during compaction while the compressibility is influenced by all factors affecting these three parameters. In particular, the compact size or aspect ratio strongly affects the amount of friction at the die walls and therefore the compressibility while the compactability is on the contrary independent of the compact aspect ratio [10].

All these characteristics are strongly affected by the lubricant/binder system. Lubricant is required to reduce the friction at die walls and ensure a good transfer of the compaction force throughout the part, low ejection forces and good surface finish and minimize tool damage. Lubricant is also needed to reduce internal friction between particles. On the other hand, the pore free density, which determines the upper limit that can be achieved during compaction, is mainly affected by the amount of low-density additives such as lubricant, binder and graphite. Increasing their contents in the mix significantly reduces the pore free density, and thus the achievable green density at high pressures. As a rule of thumb, each addition of 0.1% lubricant/binder decreases the pore free density by about 0.05 g/cm³. In the case of high densification processes such as warm compaction, the amount of organic materials should be kept as low as possible. An addition of 0.6% lubricant, suitable for warm compaction, appears to be a good compromise between the pore free density and the lubrication of die walls.

ANALYSIS OF THE COMPACTION PROCESS

The compaction process in a rigid die can be described by two fundamental parameters: The slide coefficient, which gives an evaluation of the level of friction between powder particles and die walls, and the compactability, which measures the intrinsic reaction of a powder to an outside pressure [10]. The slide coefficient and the compactability are defined below.

Gasiorek and al. [11,12] have introduced an empirical relation for the determination of a slide coefficient η on a single action press. The slide coefficient η characterizes the efficiency of transferring the compaction force throughout the part and the densification uniformity. η is given by the following equation,

Equ (1):
$$\boldsymbol{h} = \left(\frac{Pt}{Pa}\right)^{\left[\frac{4F}{SH}\right]}$$

where Pa is the pressure applied to the compacting punch, Pt the pressure transmitted to the stationary punch, F the cross-section area, S the cross-section perimeter, H the height.

The factor 4F/SH represents the compact aspect ratio or compact geometry factor. For a cylindrical compact, the factor 4F/SH is equal to D/H where D is the diameter of the compact. η can vary between 0 and 1, 0 representing an infinite friction and 1 no friction. The higher the η ,

the lower the friction loss and the better the lubrication and densification uniformity. For a given IN die density, the value of the slide coefficient proved to be a good parameter to compare the lubrication behavior of similar steel powder mixes containing different types of lubricants [13]. However, the value of slide coefficient is far from being constant through the pressing process. The variation of the slide coefficient results in fact from the complex evolution of the friction coefficient and the angle of pressure transmission or radial to axial stress ratio. The evolution of the coefficient of friction and the stress ratio during compaction is discussed in details in reference 14.

On the other hand, as mentioned earlier, the compactability is defined as the intrinsic ability of a powder to be densified in the absence of friction at die walls. The compactability is by definition independent of the compact aspect ratio unlike the compressibility as defined earlier. It can be expressed by the relation between the average IN die density and the average pressure seen by the compact. Considering that the density varies linearly along the compaction axis as shown by several researchers [15,16], it can be stated that the density at mid-height is equal to the average density. Therefore, the average pressure or net pressure, P_{NET} can be evaluated at mid-height of compact with the following equation for a cylindrical compact,

Equ. (2):
$$P_{NET} = Pa * h^{\left(\frac{H}{2D}\right)} = (Pa * Pt)^{1/2}$$

EXPERIMENTAL PROCEDURES

Lab scale mixes made of ATOMET 1001 + 2.5% Ni + 0.6% graphite + 0.6% lubricant were prepared with different high melting point lubricants: Li stearate, EBS wax and a special lubricant named WP for the purpose of that study. These mixes were binder treated to improve mix flowability and homogeneity. The level of lubricant was 0.6%.

The compaction and ejection characteristics of mixes were determined with an instrumented single action compacting press known as the Powder Testing Center (PTC) [10]. This lab press allows continuous recording of the moving punch displacement and pressures applied to the moving punch and transmitted to the stationary punch all along the compaction and ejection process, allowing determination of the compactability, slide coefficient and the ejection forces. A more complete description of the equipment and methodology used can be found in references 10 and 13. Cylindrical specimens with a height of either 8 or 15 mm were pressed in a D2- high speed steel die having a diameter of 9.525 mm at a compacting rate of 1 mm/sec. At least 5 specimens were pressed for each material and condition. The compacting temperature was varied from 90 to 150°C. It is worth mentioning that the aspect ratio of the 8 and 15 mm compacts pressed in the PTC are respectively 2.4 and 4.5 times higher than that of standard ¹/₄ in thick T.R.S bars normally pressed in lab presses. In addition, the compacting rate is also much higher than in lab presses. Those compacts are therefore more representatives of parts commonly produced at production scale. Most of the specimens were pressed to an IN die density of 7.25 g/cm³.

VALIDITY OF THE METHOD USED TO DETERMINE THE COMPACTABILITY

In this study, the compactability or the intrinsic ability of a powder to be densified in the absence of friction at die walls, is given by the relation between the average IN die density and the net pressure measured at mid-height of the compact according to equation 2. It should be independent of friction, and thus, specimen size or aspect ratio. Therefore, to validate this statement and confirm that the compactability as expressed in this study only reflects the intrinsic mechanical behavior of the powder, compacts having different heights were pressed in an instrumented press called PTC. Mix containing lubricant WP was used.

Figure 1 shows the relation between the IN die density and, a- the applied pressure and, b- the net pressure for two different compact heights. It is seen in Figure 1a that the applied pressure needed to reach a given IN die density was higher for the compact having the highest aspect ratio. The increase in pressure is directly related to the higher amount of friction at the die walls for the tallest compacts. Therefore, the applied pressure can not be used to determine the intrinsic law of densification of a material. However, it is seen in Figure 1b that the densification curves are practically identical for both specimen heights when net pressure is used. It can be concluded that the net pressure as measured by equation 2 is independent of friction and compact aspect ratio and that the relation between density and net pressure provides a very good measurement of the powders intrinsic compactability.



Figure 1. Relation between the IN die density and the compacting pressure for two different part heights. a) Density vs applied pressure, b) Density vs net pressure.

RESULTS AND DISCUSSION

Table 1 summarizes the characteristics of specimens pressed to an IN die density of 7,25 g/cm³ as determined with the instrumented lab press called PTC. The compactability is expressed in this table by the net pressure needed to reach an IN die density of 7,25 g/cm³. The compaction characteristics, compactability, slide coefficient, springback and green density and the ejection characteristics of mixes are discussed in the following paragraphs.

1. Behavior of Mixes During Compaction

Figure 2 shows typical densification curves at 90 and 150°C for all lubricants tested. It is seen in Figure 2a that Li stearate had the best compactability all along the compaction process at 90°C while EBS wax and lubricant WP showed quite similar densification curves. EBS wax was however slightly more compressible than lubricant WP at high net pressure. However, at 150°C, as seen in Figure 2b, lubricant WP had a better compactability than the other mixes when the net pressure exceeds 25 tsi. Both mixes containing EBS wax and Li stearate showed very similar compactability curves at high pressures. For net pressures lower than 25 tsi, mixes containing Li stearate and lubricant WP showed very similar compactability characteristics.

The capability of a lubricant for warm compaction is typically measured at high densities or high pressures since it is under those conditions that mixes will be used. Figure 3 shows the effect of compacting temperature on the net pressure needed to reach an IN die density of 7.25 g/cm³ for these lubricants. Different trends were observed. It is seen for EBS wax that the net pressure did not vary significantly between 90 and 150°C while it increased slightly with Li stearate. This indicates that no gain in compactability is obtained with these lubricants when temperature is

Mix	Temp.	Comp. Pressure, tsi		Density, g/cm ³		Volume	Volume Slide	Ejection Pressure, tsi	
FN-0205	°C	Applied	Net	IN Die	Green	Change, %	Coefficient	Stripping	Sliding
	90	47.5	38.6	7.249	7.160	1.25	0.614	3.01	1.48
EBS wax	115	47.0	37.8	7.247	7.174	0.98	0.604	3.34	1.45
	130	48.1	39.0	7.252	7.213	0.67	0.618	3.22	1.33
	150	46.5	38.1	7.247	7.185	0.87	0.651	3.59	1.19
	90	45.9	37.4	7.249	7.131	1.56	0.612	2.48	1.47
Li Stearate	115	47.9	38.6	7.252	7.176	1.06	0.597	2.66	1.60
	150	49.3	39.1	7.248	7.205	0.60	0.579	2.91	1.59
	90	45.8	39.5	7.247	7.167	1.22	0.696	1.93	1.05
WP lub	115	43.5	37.3	7.245	7.172	1.02	0.692	1.96	1.01
	130	43.4	36.1	7.245	7.195	0.66	0.661	2.39	1.11
	150	42.8	36.0	7.251	7.211	0.56	0.676	2.39	1.08

 Table 1. Results of tests carried out on the instrumented lab press PTC.



Figure 2. Typical densification curves obtained with different lubricants at a) 90°C, b) 150°C.

increased. However, a different trend was obtained with lubricant WP. Indeed, increasing the temperature up to 130°C significantly improved the compactability as shown by the reduction of the net pressure. However, a rise of the temperature from 130 to 150°C did not lead to a further improvement of the net pressure, which remained similar to that obtained at 130°C. The net pressure was about 1 to 3 tsi lower with lubricant WP than with the two other lubricants between 115 and 150°C. Therefore, the use of lubricant WP allowed therefore to obtain a steel powder mix with better intrinsic compactability than the other lubricants within this temperature range.

The slide coefficient is the second key factor to consider during compaction. Figure 4 shows typical curves of slide coefficient as a function of IN die density at 90°C. It is worth mentioning that very similar curves are obtained when comparing the slide coefficient to the net pressure. It is clearly seen that the slide coefficient was higher at any density with lubricant WP. The same behavior was obtained for all lubricants and at temperatures investigated. This behavior was also obtained when pressing at higher IN die density or net pressure. At the beginning of compaction, the slide coefficient decreased due to the increase of the radial to axial stress ratio. However, above about 6.0 g/cm³, the slide coefficient increased continuously with density. Since the stress ratio tends to reach a constant value when pressure reached a certain level, the continuous rise of the slide coefficient as density increased is mainly caused by a decrease of the friction coefficient. This may be partly due to a higher amount of lubricant expelled out of the compact towards the die walls as density increased. However, other factors such as the reduction of the specimen surface roughness, the increase of the effective surface area to carry the radial stress and the effect of pressure and temperature on the viscosity of lubricant, are likely playing key roles in the increase of slide coefficient as density increased. [13].

Figure 5 shows the variation of the slide coefficient as obtained at 7.25 g/cm³ as a function of the compacting temperature for all lubricants. Again, as was the case for the compactability, significant differences were seen between lubricants. In the case of EBS wax, the slide coefficient remained at about 0.61 between 90 and 130°C and rose to about 0.65 at 150°C. With Li stearate, the slide coefficient decreased slightly with temperature for the entire range investigated from about 0.62 to 0.58. Finally, much higher slide coefficients were measured with lubricant WP. The slide coefficient varied between 0.66 to 0.70 for the entire temperature range,







Figure 4. Typical curves of slide coefficient as a function of IN die density at 90°C.

the maximum value being obtained at 90 and 115°C. It is worth mentioning that the slide coefficient obtained at 90 and 115°C was similar to that measured at room temperature, clearly indicating that the lubricating properties of lubricant WP are maintained when moderately heated. This was not the case for the two other lubricants.

The rise in slide coefficient observed between 130 and 150°C for EBS wax is not believed to be related to an improvement of the friction properties of that lubricant. In fact, the use of EBS wax resulted in a lot of noise and stick and slip at 150°C. The increase in slide coefficient is believed to be linked to an increase in the amount of lubricant at the die walls. Indeed, EBS wax melts at 145°C and it is possibly much easier to expel out of the compact at 150°C.

The higher slide coefficient obtained with lubricant WP will result in a much better transfer of the pressure throughout the part and a lower loss of pressure due to friction during compaction. The pressure loss between the top and bottom punches was between 2 to 5 tsi higher for EBS wax and Li stearate as compared to lubricant WP between 90 and 150°C.

The combined effect of slide coefficient and compactability on densification is measured by the applied pressure needed to reach a given IN die density. Figure 6 illustrates the variation of the applied pressure needed to reach a 7.25 g/cm³ IN die as a function of the compacting temperature. Basically, the same trends as those observed with the net pressure are shown. However, the difference in pressure between lubricant WP and the others is incremented by about 2 tsi due to differences in slide coefficient. It is seen that lubricant WP is superior to the other lubricants for the entire temperature range, even at 90 and 115°C despite the fact that the compactability of lubricant WP was respectively worst or only slightly better (see Figure 3).

Figure 7 illustrates the effect of part height on the applied pressure based on results obtained at 115°C. For all lubricants, the applied pressure must be increased when part height is increased. However, the increase is more important for lubricants having poor slide coefficient such as EBS wax and Li stearate than in the case of lubricant having better slide coefficient such as lubricant WP. This emphasizes again the importance of efficiently transferring the pressure throughout the part, especially for parts having high aspect ratios such as those commonly manufactured in the P/M industry.



Figure 5. *Effect of compacting temperature on the slide coefficient measured at* 7.25



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Based on results shown in Figure 6, the optimum temperature range for lubricant WP is between 115 and 130°C. However, It should be considered that the high compaction rate used in a

production press leads to a significant increase in the temperature of the part, which is besides proportional to the part height. This aspect has to be considered when setting the tool and powder temperature.

2. Part Expansion and Green Density

The volume expansion that parts undergo at ejection is also a key factor to consider when studying the compaction process since it determines, in conjunction with the slide coefficient and the compactability, the green density that will be achieved for a given applied pressure, part geometry, size and tooling. Figure 8 shows the effect of compacting temperature volume on expansion and green density. Increasing the temperature led to a significant reduction of the volume expansion for the different lubricants. This reduction in volume expansion is mainly related to a reduction of the springback parallel to the compaction axis temperature increased. as The radial springback acting perpendicularly to the compaction axis changed only slightly with temperature.

The drop of the axial springback is thought to be mainly related to the softening of steel particles which resulted in a larger amount of plastic deformation, metal to metal contact and cold welding between steel particles as shown by Gagné [5]. This likely contributed to increase the effective cross section area and reduce therefore the effective stress in the part. Gagné also showed that the amount of lubricant lying between steel particles is reduced when temperature increased. However, it should be emphasized that a different trend can be obtained at higher compaction rate under certain conditions as observed on a production press [7].



Figure 7. Effect of part height on the applied pressure needed to reach an IN die density of 7.25 g/cm³.



Figure 8. Effect of compacting temperature on the volume expansion at ejection and green density for specimens pressed to 7.25 g/cm³ IN die.

The reduction of the volume expansion with temperature resulted in a significant increase in green density for all the lubricants. For example, the green density increased from 7.16 to 7.21 g/cm³ when raising the compacting temperature from 90 to 150° C with lubricant WP. It is interesting to note that a gain in green density was obtained with Li stearate and EBS wax even if these lubricants showed no improvement in compactability and slide coefficient as temperature increased. This clearly shows the importance of properly characterizing the compaction process to have a better understanding of the factors affecting the final density.

The relation between the green density and the applied pressure, which is known as the compressibility, is in fact the result of the combined effect of the slide coefficient, the compactability, the springback and compact aspect ratio. The effect of compacting temperature on the compressibility of cylindrical specimens given by the applied pressure needed to reach a green density of 7.20 g/cm³ is shown in Figure 9. It is seen for all the mixes that increasing the temperature led to a reduction of the applied pressure and therefore to an increase of the compressibility. However, lubricant WP remained the most compressible lubricant for the entire temperature range while Li stearate was the worst.

3. Ejection

Figure 10 shows typical ejection curves obtained at 115°C. Curves were intentionally shifted along the "x axis" to allow a better discrimination of curves. It is seen for lubricant WP and Li stearate that the ejection was smooth, indicating a good integrity of the lubricant film at die walls during ejection [13]. However, the ejection curves obtained with EBS wax were much more "noisy", important jumps and drops in pressure being observed all along the ejection. This type of behavior is indicative of an important "stick and slip" phenomenon due to intermittent disruptions of the lubricant film [13]. Increasing the temperature was found to increase the "stick and slip" phenomenon for all the lubricants. However, this phenomenon was more important with EBS wax than with the other lubricants.







Figure 10. Typical ejection curves obtained at 115°C for specimens pressed to 7.25 g/cm³ IN die.

Figure 11 shows the stripping and sliding pressures as a function of the temperature. The sliding pressure is the pressure averaged over the first 2.54 mm or 0.1 po of travel after the peak. As it was the case during compaction, lubricant WP showed the best lubricity for the entire temperature range studied, the stripping and sliding pressures being significantly lower with WP

than with the other lubricants. For example, the stripping pressure was about 50% and 25% lower with WP than with EBS and Li stearate respectively. As a result, the surface finish of compacts was much better In all cases, increasing the with WP. temperature led to an increase of the stripping pressure. However, it did not exceed 2.4 tsi with lubricant WP. The sliding pressure remained stable when temperature increased with WP and Li stearate while it decreased with EBS wax. The sliding pressure was even lower with EBS wax than with Li stearate for temperatures equal or higher than 115°C. However, EBS wax caused significant "stick and slip" during ejection.

CONCLUSIONS

The densification and ejection characteristics of FN-0205 mixes containing 0.6% lubricant were studied with an instrumented laboratory press at different

Figure 11. Effect of compacting temperature on the stripping and sliding pressure for specimens pressed to 7.25 g/cm³ IN die.

compacting temperatures ranging between 90 and 150°C, leading to the following conclusions:

- The instrumented lab press used was found to be an efficient technique to evaluate the compaction and ejection processes. In particular, it allowed to quantitatively characterize the key phenomena controlling compressibility, i.e. the friction at die walls, the compactability and the springback.
- EBS wax and Li stearate provided a poor lubrication of die walls when heated. In addition, no gain in compactability was obtained by increasing the temperature with these lubricants.
- A lubricant named WP was found to provide a much better lubrication of die walls during compaction and ejection and a better compactability than EBS wax and Li stearate. As a result, the applied pressure needed to reach a given density and the ejection force were considerably reduced and the surface finish of specimens improved.
- The optimum compacting temperature of this new lubricant appeared to be between 115 and 130°C. No gain in compactability and lubricity at die walls is obtained above 130°C.
- The capacity to reduce the friction at die walls is a key factor to consider when selecting a lubricant/binder system for warm compaction. This factor controls both the efficiency of transferring the pressure throughout the part as well as the surface finish. It determines therefore the minimum amount of lubricant that should be added to the mix.





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