



**INFLUENCE OF POWDER MIX FORMULATION ON GREEN AND SINTERED
PROPERTIES OF WARM PRESSED SPECIMENS**

by

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INFLUENCE OF POWDER MIX FORMULATION ON GREEN AND SINTERED PROPERTIES OF WARM PRESSED SPECIMENS

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ABSTRACT

Warm compaction is a technique, which enhances the green density of pressed components by heating a powder mix and tooling to temperatures ranging typically between 90 and 150°C. The gain in density achieved by warm pressing compared to “cold” compaction is typically from 0.07 to 0.30 g/cm³ depending on the size of parts, the powder formulation and the compaction parameters. However, to take advantage of this beneficial effect of a moderate increase of the compacting temperature on densification, powder mixes must be properly designed to provide good and stable feeding of the die cavity at the processing temperature to allow consistent part to part weight and density. Also, the base powder and the admixed elements must be properly selected to maximize both the green and sintered densities while maintaining low ejection forces and good part surface finishes. In particular, the selection of the lubricant is key in warm compaction.

This paper discusses the behavior of various powder mixes warm pressed under different compacting conditions. The influence of base steel powder grade and the amount and type of various additives on the green and sintered properties is presented. Particular attention is paid to the influence of lubricant on green density and ejection forces.

I. INTRODUCTION

Achievement of high density at reasonable cost is one of the major objectives of the P/M industry for the production of P/M components requiring high static and dynamic properties. In cold compaction, the maximum green density that can be achieved is usually in the range of 7.05 to 7.25 g/cm³ depending on the compressibility of the base powder, the amount of additives and lubricant, the part geometry and height, and the compacting conditions. On the other hand, the double press/double sintering (DPDS) and forging techniques can be used to achieve much higher density, typically in the range of 7.4 to 7.5 g/cm³ for the DPDS process and to nearby full density for the forging process [1]. However, the production costs associated with these

techniques are quite significant. The warm pressing process, which consists of pressing a preheated powder mix in a heated die [2], is a very interesting alternative avenue allowing the achievement of higher density compared to cold compaction at a lower cost than the DPDS and forging techniques. Typically, final densities in the range of 7.25 to 7.45 g/cm³ can be achieved after a single press/single sinter step with warm pressing [3]. Even higher densities can be achieved by using very elevated compacting pressure and/or high temperature sintering [4] or by combining the warm compaction and die wall lubrication techniques.

However, the production of high performance parts requires a good understanding of the phenomena taking place during the compaction, ejection and sintering stages in order to optimize the sintered density and the static and dynamic properties. In particular, the material formulation, especially the steel powder grade and the type and level of the admixed lubricant are key factors controlling the densification and ejection performance of warm pressed materials. Also, the sintered density is not solely a function of the density achieved after compaction but also strongly depends on the degree of shrinkage during sintering. In particular, the final density is very sensitive to the amount and type of admixed elements used to improve the strength of the P/M components. The objective of this paper is to review the critical factors affecting the green and sintered characteristics of sintered components. The effect of the compacting temperature, base powder, low-density additives such as graphite and lubricant as well as of admixed elements like copper and nickel is discussed. Particular attention is paid to the role of lubricant in warm compaction and to its influence on the green and ejection characteristics of FLOMET WP steel powder mixes.

II. FACTORS AFFECTING DENSIFICATION

Compressibility

The green density that can be achieved in a closed die is a function of numerous factors related to the compaction process and part characteristics (compacting conditions, tool materials, clearance and design and part shape, size and complexity) and the steel powder compressibility. The compressibility defines the capability of a powder mix to be densified. It represents the relation between the green density and the applied pressure. More specifically, the compressibility of a material 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 [5]. However, the compressibility is a function of three key characteristics or phenomena that take place during the compaction and ejection processes [6]:

- 1- the compactability which defines the intrinsic ability of a powder to be densified in the absence of friction at die walls,
- 2- the friction between the compact and die walls,
- 3- the springback or volume expansion at ejection.

All these phenomena and properties can be measured with an instrumented laboratory press such as the PTC (Powder Testing Center) [7]. Thomas et al proved that such an instrumented press can be used to evaluate the behavior of different lubricants [8]. The friction at die walls can be expressed by the slide coefficient during compaction and the stripping and sliding forces during ejection. It should be emphasized that the amount of friction at die walls during the compaction

process as well as the springback are dependent on the compact size or aspect ratio while the compactability is not [7]. Therefore, the compressibility of a powder is strongly affected by the part aspect ratio and usually decreased as the part size increases.

Effect of Compacting Temperature

It is known that a moderate increase of the temperature up to about 150°C lowers the yield strength and increases the ductility and malleability of steel particles [9, 10]. As a result, a moderate increase in the compacting temperature is beneficial to the compressibility as illustrated in Figure 1. Indeed, it is shown that increasing the compacting temperature from 20 to 150°C results in a gain in green density of about 0.15 and 0.13 g/cm³ at 40 and 50 tsi respectively for a FN-0205 mix pressed using die wall lubrication without internal lubricant. It should be noted that the relative gain in density obtained by raising the compacting temperature decreases as the compacting pressure increases. For example, the gain in density achieved by raising the temperature from 20 to 150°C would be about 0.17 g/cm³ at 30 tsi compared to 0.10 g/cm³ at 70 tsi. This is explained by the fact that density levels off at high compacting pressure.

Raising the compacting temperature does not only affect the steel powder ductility but also affects the volume expansion of parts at ejection. Indeed, it was demonstrated on an instrumented laboratory press that increasing the compacting temperature did contribute to the reduction of the volume expansion of the part at ejection and, therefore, to an increase of the green density as shown in Figure 2 [6]. However, it should be pointed out that a different behavior could be achieved on a production scale depending on the compacting conditions and part size [11].

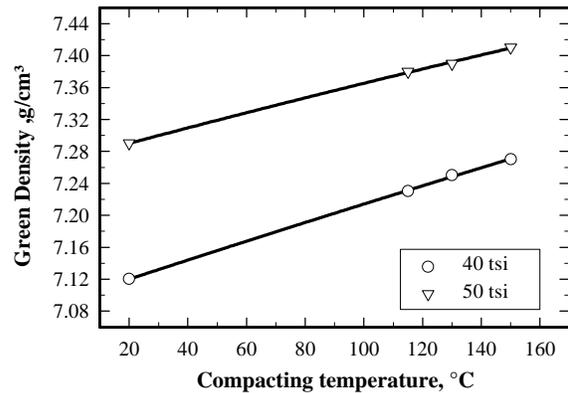


Figure 1. Effect of compacting temperature and pressure on green density of specimens pressed with a mix of ATOMET 1001+0.6% grap.+2.5% Ni, using die

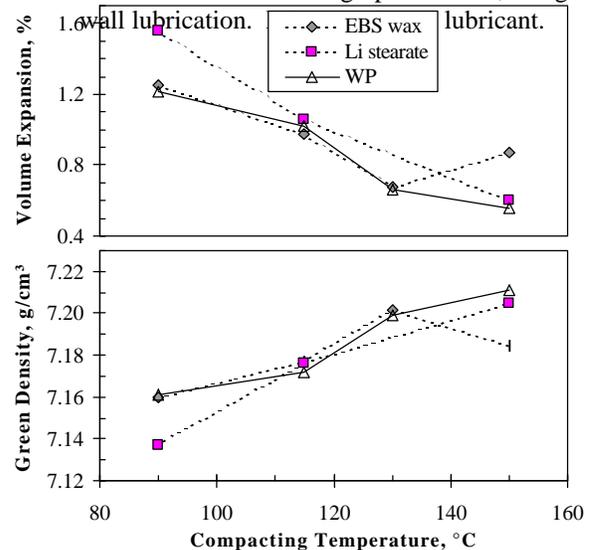


Figure 2. Effect of compacting temperature on the volume expansion at ejection and green density for specimens pressed to 7.25 g/cm³ IN-Die.

Effect of base material

Steel powders are usually selected as base material for high-density applications because of their higher compressibility compared to iron and sponge powders. However, a wide range of steel powder grades with different types and levels of alloying elements added either to the melt before atomization (pre-alloyed steel powder) or during the annealing treatment (diffusion-bonded powder) are available in the market depending on the hardenability required. Also, alloying elements can be admixed to the base steel powder to increase the hardenability. It is well known that these powders exhibit significant differences in compressibility. Figure 3 gives the compressibility curves of different steel powder grades at room temperature. It is seen that unalloyed steel powders such as the ATOMET 1001HP and ATOMET 1001 exhibit better compressibility than low alloyed grades such as ATOMET 4601, but lower sintered hardness and strength. In fact, raising the concentration of alloying elements increases the hardness of the steel particles, which in turn deteriorates the powder compressibility [12]. In that sense, molybdenum is a very interesting pre-alloying element since it is very efficient in increasing strength with only a minor effect on powder compressibility as shown in Figure 3 [13]. This is the reason why molybdenum low alloy steel powders such as ATOMET 4401 are widely used in order to achieve high green density after compaction and high strength after sintering. Also, elements such as Ni and Cu can be either admixed or diffusion-bonded to increase the hardenability and strength of P/M components while maintaining compressibility.

The use of warm compaction and a special lubricant/binder specifically designed for that purpose appears to be particularly interesting for low alloyed steel powders having high hardenability but low compressibility at room temperature. Indeed, Figure 4 shows the effect of increasing the compacting temperature from 20 to 130°C and using a special lubricant (WP) on the densification for steel powder grades having different hardenability (steel + 0.6% graphite + 0.5%lub). A gain in density of 0.29 g/cm³ at 45 tsi was obtained with ATOMET 4701 compared to 0.13 and 0.15 g/cm³ for the

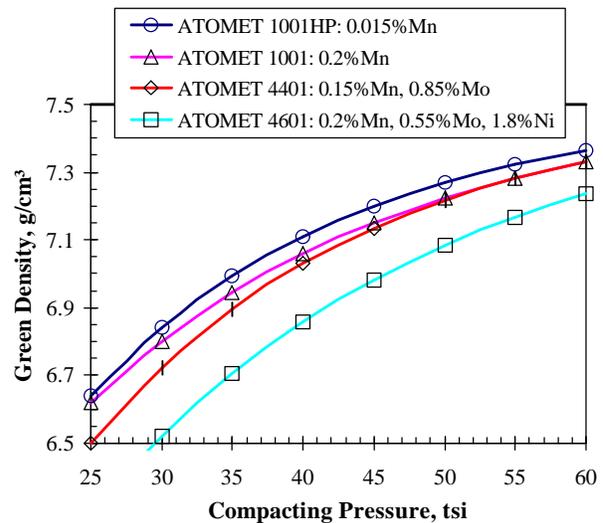


Figure 3. Compressibility curves of steel powders having various concentration of pre-alloyed elements at room temperature (0.5% Lub)

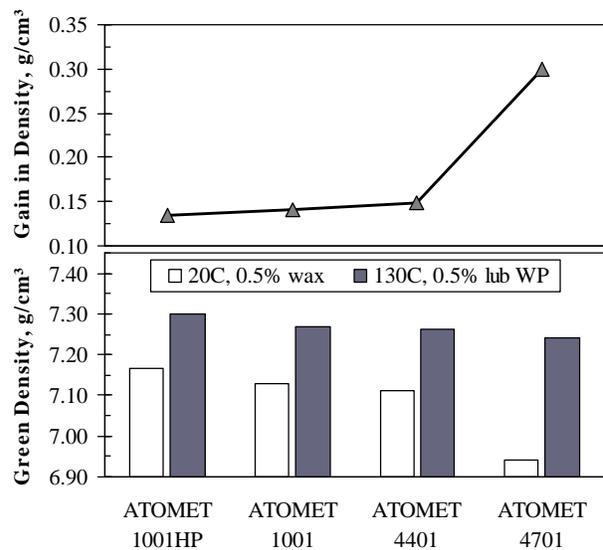


Figure 4. Effect of compacting temperature and lubricant type on green density and the gain in density of various steel powders (Steel + 0.6% Grap. + 0.5% Lub, pressed at 45 tsi)

ATOMET 1001HP and 4401 mixes respectively. In fact, the green density reached at 130°C and 45 tsi with the ATOMET 4701 mix was almost identical to that of the other mixes, i.e. about 7.23 g/cm³ compared to a maximum of 7.30 g/cm³ reached with the ATOMET 1001HP mix. It is therefore possible by using warm compaction to achieve very good green density with pre-alloyed steel powder having relatively high hardenability, thus allowing achievement of very good sintered properties.

Effect of lubricant and binder

As is the case for cold compaction, lubricant is added to mixes mainly to ease the compaction and ejection processes. It must also provide good flow. Also, addition of a binder is necessary in warm pressing mixes to improve the homogeneity, flow and robustness of mixes and the part-to-part consistency [14]. These additives have very low specific gravity compared to steel (about 1 g/cm³ versus 7.86 g/cm³). Therefore, increasing the lubricant/binder content in the mix significantly reduces the pore free density, or the theoretical maximum density that can be reached if all the porosity is eliminated. This is well illustrated in Figure 5 which clearly shows that increasing the lubricant content reduces the green density at high compacting pressure. Typically, each addition of 0.1% lubricant/binder decreases the pore free density, and thus the maximum achievable green density by about 0.05 g/cm³. However, at relatively low compacting pressures, increasing the lubricant content is beneficial to the green density. In fact, at low relative density, lubricant improves the particle rearrangement and the transfer of the compacting pressure throughout the part.

The amount of lubricant in the mix is even more important in warm compaction than in cold compaction due to the higher densities reached for a given compacting pressure. Indeed, densities up to 98 to 98.5% of the pore free density can be achieved by warm compaction at about 50 tsi. It is worth mentioning that the maximum green density achievable by warm compaction is limited to about 98.5% of the pore free density. The volume expansion of the part during ejection explains mainly why the green density is limited to such a level [6, 11]. Therefore, the amount of lubricant and binder should be kept as low as possible in warm pressing mixes. However, reducing the lubricant content in the mix would also reduce the amount of lubricant migrating to die walls during

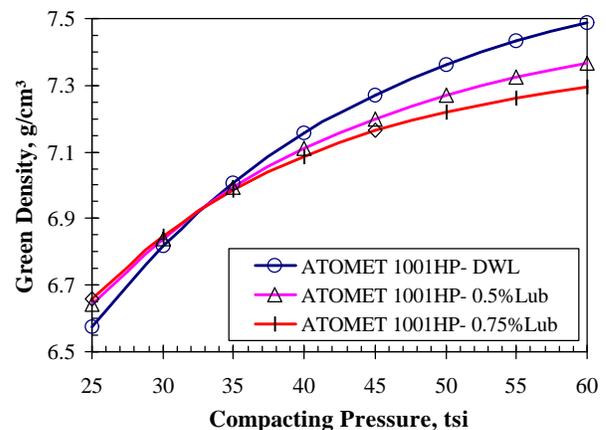


Figure 5. Effect of lubricant content on the compressibility of ATOMET 1001HP at room temperature.

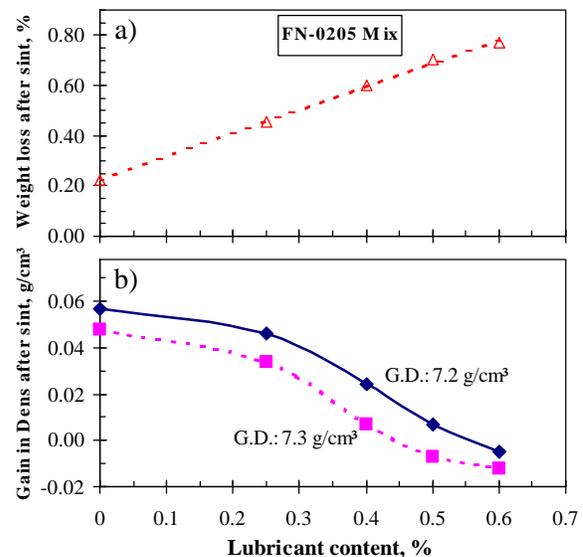


Figure 6. Effect of the lubricant content on a) weight loss after sintering and, b) gain in density after sintering (FN-0205 mixes).

compaction. Therefore, the lubricant must be very efficient in order to maintain a good lubrication of die walls, especially when densities of 7.30 g/cm³ or higher are targeted. This is described in greater detail later.

Finally, even if increasing the lubricant content is beneficial to the green density at relatively low compacting pressures, it should be remembered that the lubricant is burnt off during the sintering operation. The higher the lubricant content, the higher the weight loss after sintering as shown in Figure 6a. Because of this, the lubricant content also adversely affects the gain in density after sintering as shown in Figure 6b. For example, the sintered density of a FN-0205 mix will be respectively 7.25 and 7.21 g/cm³ at 0.25 and 0.5% lub content for parts pressed to 7.2 g/cm³. It should also be noted that the gain in density during sintering decreases as the green density increases.

Effect of graphite

Graphite is added to steel powder to increase the strength of P/M components after sintering. However, as it is the case with lubricant, graphite has a relatively low specific gravity (~2.3 g/cm³) and reduces the pore free density. Figure 7 shows the effect of the graphite content on the compressibility of mixes made of ATOMET 4401, 1%Cu and 0.75% Zn St. Each addition of 0.1% graphite decreases the pore free density and thus, the maximum green density achievable by about 0.02 g/cm³. However, at relatively low compacting pressures, increasing the graphite content has only a minor effect on green density. In fact, considering that the pore free density is reduced when graphite is increased, it can be seen that graphite, which is known as a good solid lubricant, is beneficial to the densification.

Influence of Cu and Ni

Cu and Ni are very popular additives admixed to the steel powder mixes to improve the strength and mechanical properties of P/M components. Such high-density additives have only a minor effect on the pore free density and green density. However, they have a significant effect on the densification process during sintering. Copper melts at 1085°C and penetrates the iron grain boundaries while nickel diffuses in the solid state at a conventional sintering temperature of

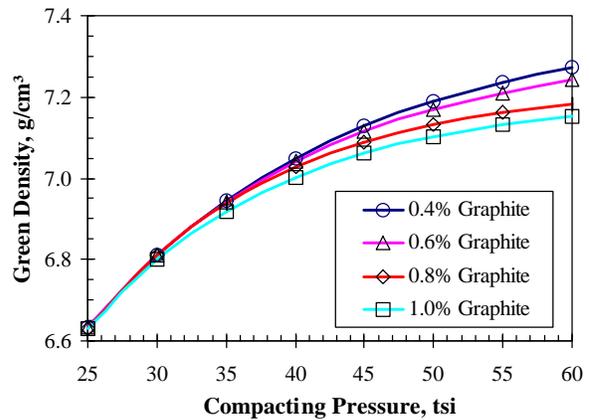


Figure 7. Effect of the graphite content on the compressibility at room temperature of mixes made of ATOMET 4401+ 1%Cu + C + 0.75% Zn St

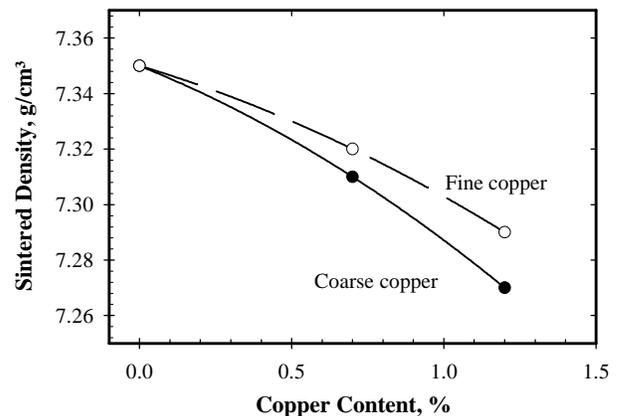


Figure 8. Effect of copper content on the sintered density of TRS specimens pressed at 130°C and 45 tsi (ATOMET 4401+3.5% Ni+0.3% C+Cu+0.5% Lub; pressed to 7.35 g/cm³).

1120°C. In fact, Ni requires higher sintering temperatures to fully diffuse in the iron particles [15, 16]. These differences in the behavior and the diffusion mechanisms explain why these elements have a different effect on density. Indeed, copper additions induce growth when melting at 1085°C and lower the sintered density, particularly for low graphite mix formulations, as illustrated in Figure 8 for FLOMET WP mixes made of ATOMET 4401-3.5%Ni-0.3%C-0.5%lub. However, the use of fine grade of copper induces less growth and gives higher sintered density than coarser grades. On the other hand, the addition of nickel, which is an austenite stabilizer, promotes shrinkage during sintering and reduces the expansion during the $\gamma \rightarrow \alpha$ phase transformation. As a result, Ni increases the sintered density as shown in Figure 9. Each increment of 1% Ni results in an increased density of about 0.01 g/cm³.

III. ROLE OF LUBRICANT

Influence of Lubricant Type

As indicated previously, the lubricant has a strong effect on the pore free density and should therefore be kept as low as possible (as well as the binder) to enable densities of 7.3 g/cm³ or higher during compaction. The lubricant should therefore be very efficient at the compacting temperature encountered in warm pressing, which is typically between 90 and 150°C. Figure 10 shows the influence of the type of high-melting point lubricant and the compacting temperature on the compactability of FN-0205 mixes as measured on an instrumented press. It should be remembered that the compactability measures the ability of a powder to be densified in the absence of friction. The compactability is expressed in Figure 10 by the net pressure required to reach an IN-die density of 7.25 g/cm³. It is seen that using Li stearate and EBS wax do not improve compactability when the compacting temperature is increased. However, a different trend is obtained with lubricant WP. Indeed, the compactability with this lubricant is significantly improved when temperature is increased up to 130°C as shown by the reduction of the net pressure. Therefore, lubricant WP should be more suitable for warm compaction.

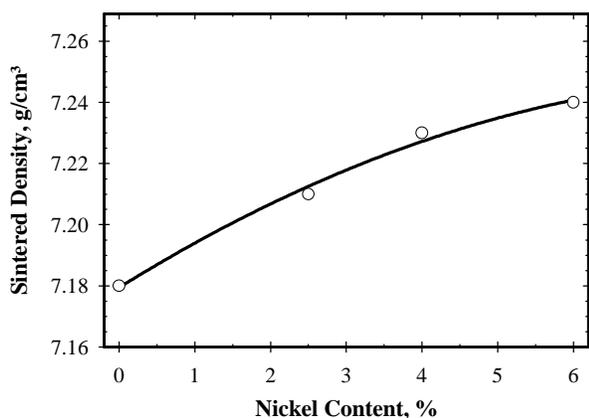


Figure 9. Effect of nickel concentration on the sintered density of TRS specimens pressed at 520 MPa and 130°C (ATOMET 4401+0.2% C+Ni+0.55% Lub; pressed to 7.20 g/cm³).

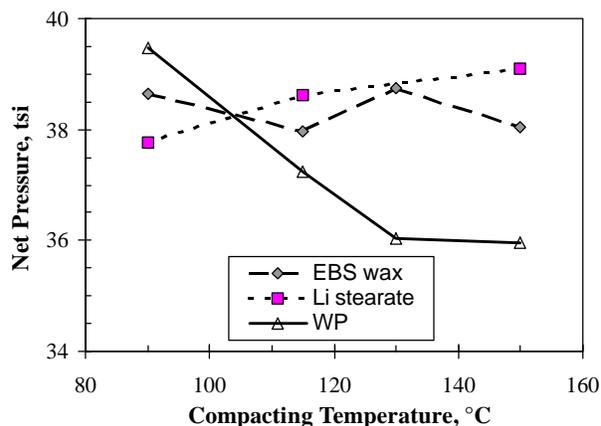


Figure 10. Influence of the lubricant type and the compacting temperature on the compactability of FN-0205 mixes, ref. . (pressed to 7.25 g/cm³ IN-Die, 0.6%Lub)

Also, it was found in a previous study relating to the behavior of powder during compaction that the capacity to lubricate and reduce the friction at die walls is the key factor to consider when selecting a lubricant/binder system for warm compaction [6]. Indeed, the lubrication at die walls controls both the efficiency of transferring the pressure throughout the part as well as the ejection forces and surface finish. Moreover, it determines the minimum amount of lubricant that could be added in the mix for a given application. Figure 11 show the influence of the compacting temperature and the type of high melting point lubricant on the lubrication at die walls during the compaction and ejection of specimens with an aspect ratio almost 2.5 times higher than that of $\frac{1}{4}$ in thick TRS bars. The lubrication during compaction and ejection is quantified by the slide coefficient and the stripping pressure (pressure needed to start ejection) respectively. It is worth mentioning that the higher the slide coefficient (which varies between 0 and 1), the lower the friction loss and the better the lubrication and densification uniformity. It is clear that the lubricant WP gives much better lubrication properties than EBS wax and Li stearate during the compaction and ejection, the slide coefficient being significantly higher while the stripping pressure remaining 25 to 50% lower than that obtained with the two other lubricants. As a result, the pressure loss throughout the part due to friction during compaction is significantly lower with lubricant WP than with the two other lubricants. This low pressure loss combined with a very good compactability (Fig. 10) contribute to obtain much better compressibility with lubricant WP when the mix is heated as shown in Figure 12. Lubricant WP is therefore a very good candidate for warm compaction applications. It is also seen that the applied pressure to reach a green density of 7.2 g/cm^3 decreases with temperature for all lubricants. However, the effect is more pronounced with lubricant WP.

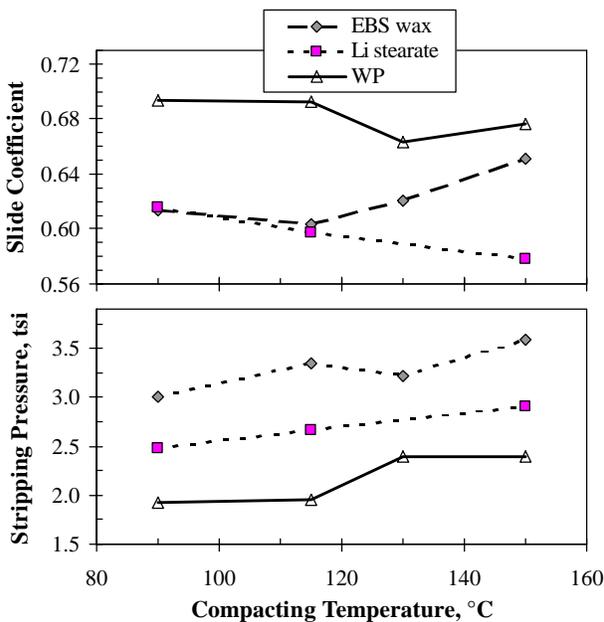


Figure 11. Effect of compacting temperature and lubricant type on the lubrication at die walls during compaction and ejection, ref (Mix: FN-0205 + 0.6% Lub, In-Die Density : 7.25 g/cm^3).

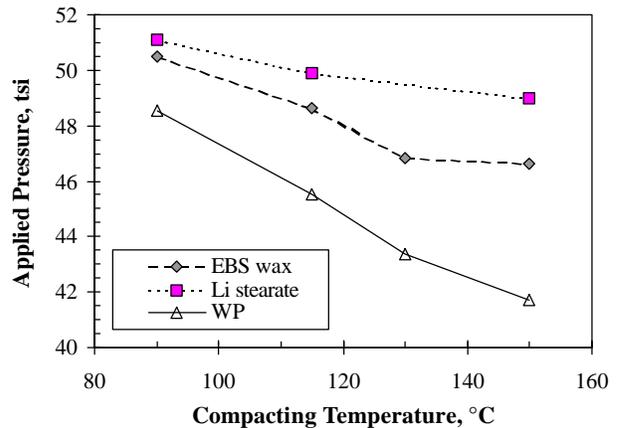


Figure 12. Effect of compacting temperature and lubricant type on the applied pressure needed to reach a green density of 7.2 g/cm^3 , ref (Mix: FN-0205 + 0.6% Lub).

Another example of the excellent lubrication properties of lubricant WP is given in Figure 13 for mixes made with ATOMET 4701, a steel powder having a much higher hardenability than the ATOMET 1001. The ejection of WP binder-treated mixes containing 0.53 and 0.60% lubricant WP pressed at 130°C is compared to that of a regular mix containing 0.75% EBS wax pressed at room temperature. It is seen that the stripping pressure measured with the mixes containing lubricant WP pressed at 130°C was significantly lower than that obtained with the regular mix pressed at room temperature even if the lubricant content was 20 to 30% lower in the WP mixes.

On the other hand, the sliding pressure, which is the average pressure measured during the ejection cycle, was similar or slightly lower with the WP mixes pressed at 130°C. In addition to that, it should be remembered that the green densities achieved by warm compaction are significantly higher. For example, the gain in density obtained at 45 tsi was about 0.29 g/cm³ as shown in Figure 5. Therefore, warm compaction is a very interesting avenue for the production of parts requiring relatively high density while maintaining excellent lubrication at die walls with steel powder having high hardenability.

Influence of Lubricant Content

The effect of the concentration of lubricant WP on the compressibility of FN-0205 mixes as measured on ¼ in. thick TRS bars at 130°C is shown in Figure 14. The mix containing no lubricant was pressed using die wall lubrication (DWL). As it is the case in cold compaction, increasing the lubricant content is beneficial to the densification at low compacting pressure. However, at high compacting pressure, the pore free density becomes critical and the density is improved by reducing the lubricant content. In fact, the density starts to level off at a lower compacting pressure when the lubricant is increased. Indeed, it is seen that the green density has almost reached its maximum at about 45 tsi for mixes containing 0.5 and 0.6% lubricant while it continues to increase at 0.25 and 0.4% lubricant. Thus, the amount of lubricant must be reduced in the mix and the compacting pressure increased in order to reach much higher density. For example, a green density of 7.40 g/cm³ is reached at 50 tsi with 0.25% lubricant versus 7.33 and 7.31 g/cm³ at 0.5 and 0.6% lubricant respectively.

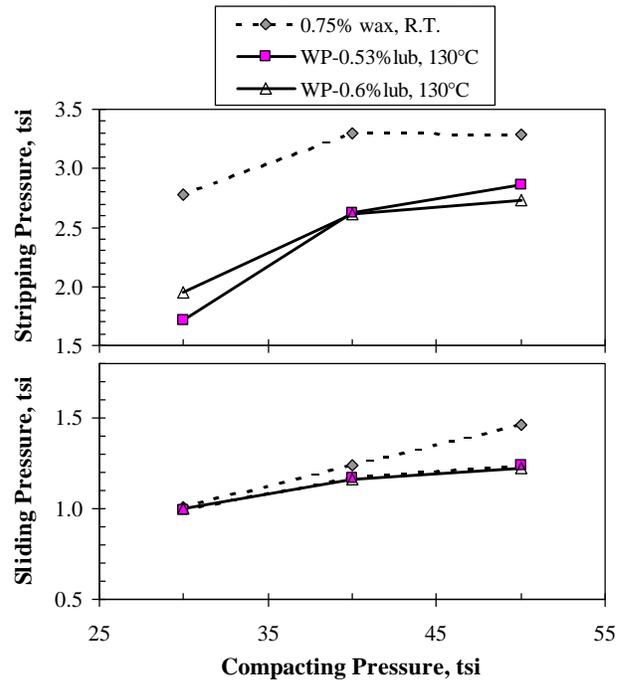


Figure 13. Stripping and sliding pressure measured at different compacting pressure with a regular mix pressed at 20°C and two warm pressing mixes pressed at 130°C. (ATOMET 4701+ 2% Cu + 0.85% C + Lub)

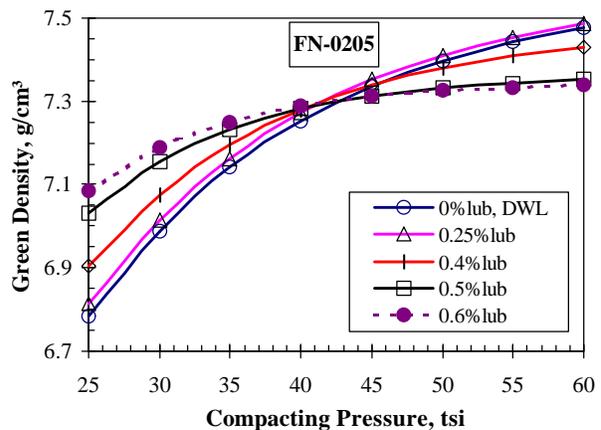


Figure 14. Compressibility curves of warm pressing FN-0205 mixes with different level of lubricant. (Compacting temperature : 130°C)

It is also interesting to note in Figure 14 that the compressibility achieved with the mix containing 0.25% lubricant (no lubricant was sprayed on die walls with this mix) was better than that achieved with the mix containing no lubricant and pressed with DWL. This result confirms that a minimum amount of internal lubricant is required to optimize the particle rearrangement and the compactability. It is worth mentioning that much higher density could be reached by using the DWL technique with a mix containing 0.25% lubricant.

As previously mentioned, the compressibility is also affected by the aspect ratio, and thus, the part size. The aspect ratio influences in conjunction with the lubricant properties the friction at die walls. Figure 15 shows the effect of part size and lubricant content on the green density obtained at 40 and 50 tsi. As expected, increasing the part thickness from ¼ to ½in. induces a reduction in green density varying between 0.03 and 0.04 g/cm³ at 40 tsi and between 0.00 and 0.03 g/cm³ at 50 tsi. The reduction in green density tends to decrease when the compacting pressure raises. Moreover, reducing the lubricant content contributes to increase the loss in density when increasing the part height. In other words, reducing the lubricant content is less beneficial on densification when part height increases. This is related to the fact that reducing the lubricant content in the mix contributes to reduce the slide coefficient and, in turn, increase the friction and the pressure loss during compaction. However, it should be noted that the density reached at 50 tsi with 0.25 % lubricant is still high at 7.38 g/cm³.

One of the major concerns when reducing the lubricant content in the mix is the ejection performance. Figure 16 shows the variation of the stripping pressure as a function of the lubricant content for ¼ and ½in. thick TRS bars pressed at 130°C and 50 tsi. As expected, decreasing the lubricant content from 0.6 to 0.25% leads to an increase of the stripping pressure from 2.3 to 3.1 tsi for ¼ in. thick bars and from 2.3 to 3.7 tsi for ½in. thick bars. A very similar trend was observed at 40 tsi, the stripping pressure being lower and the effect of reducing the lubricant content less important. In fact, decreasing the lubricant content has a more pronounced effect on the ejection when the thickness is increased. It can be seen that raising the thickness has only a minor effect on the stripping pressure at 0.5 and 0.6% lubricant but leads to an increase of the ejection force at 0.4 and 0.25%, the difference increasing as the lubricant content decreases.

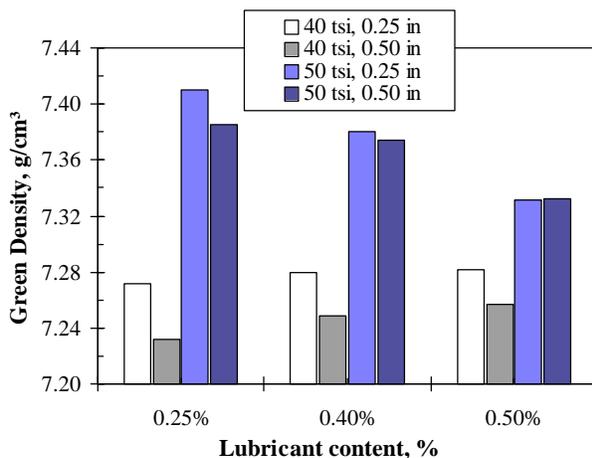


Figure 15. Influence of the compacting pressure and part height (TRS bars) on the green density obtained at 130°C with different level of lubricant (Mix: FN-0205).

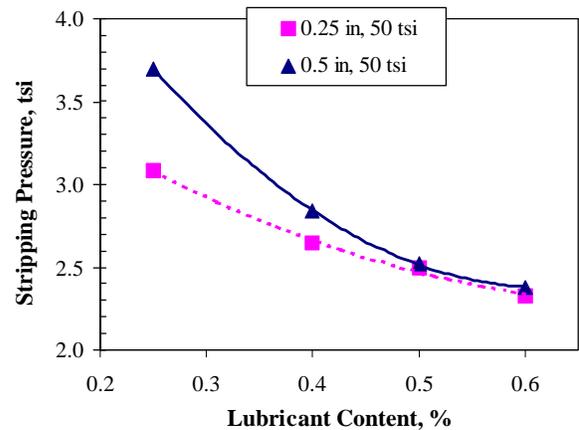


Figure 16. Effect of the lubricant content and the part height on the ejection of FN-0205 mixes pressed at 130°C and 50 tsi.

Figure 17 shows the effect of the compacting pressure and the lubricant content on the ejection of $\frac{1}{2}$ in. thick bars pressed at 130°C. The influence of the compacting pressure on the stripping and sliding pressure is strongly dependent on the level of lubricant. Indeed, at 0.25% lubricant, increasing the compacting pressure above 40 tsi leads to an increase of the stripping and sliding pressure. However, at 0.4 and 0.5% lubricant, varying the compacting pressure between 37 and 50 tsi has only a minor effect on the ejection pressure. This clearly demonstrates that lubricant WP can withstand elevated compacting pressures without losing its excellent lubrication properties. This is particularly important in warm compaction where compacting pressures are usually particularly elevated.

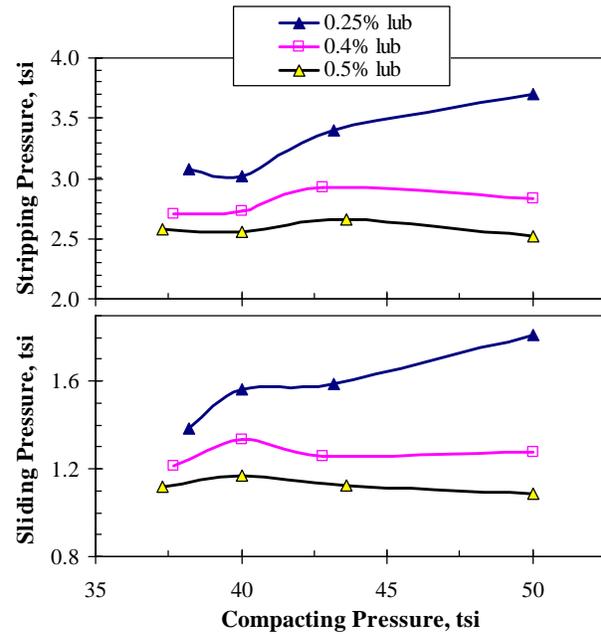


Figure 17. Influence of the compacting pressure and lubricant content on the ejection of FN-0205 mixes pressed at 130°C.

It should be mentioned that the stripping pressure obtained at 0.25% lubricant remains lower than that obtained with a mix containing 0.6% of a high-melting point polyamide wax. Indeed, the stripping pressure of that lubricant is about 4 tsi at 50 tsi and 130°C ($\frac{1}{4}$ in. thick bars). It should be noted that the optimum amount of lubricant in a mix is the one which maximizes the density while maintaining the ejection force at an acceptable level. The maximum stripping pressure that can be tolerated is a function of the tooling (die and punches), part design, density and compacting pressure.

IV. CONCLUSIONS

Optimization of density, and therefore the sintered properties by warm compaction, requires a good understanding of the effect of the base powder, admixed elements and compacting and sintering conditions in order to maximize the sintered density. The amount of admixed and/or pre-alloyed elements that reduce either the compressibility or lower the density during sintering must be carefully selected to minimize any detrimental effect of the final density. However, it was shown that using warm compaction can be particularly beneficial for steel powder grades having high hardenability but exhibiting relatively poor compressibility at room temperature. Indeed, a gain in density up to about 0.30 g/cm³ could be achieved by warm compaction with a suitable lubricant compared to cold compaction with regular lubricants for powders having high hardenability such as ATOMET 4701.

Lubricant plays a key role in warm compaction. Indeed, increasing the lubricant content reduces the maximum green density achievable by reducing the pore free density and also lowers the sintered density. The lubricant content should therefore be kept as low as possible to reach densities of 7.3 g/cm³ or higher. The lubricant selected must therefore show excellent compactability and lubrication characteristics at the temperature encountered in warm

compaction. Lubricant WP improves significantly the compactability and reduces the friction at die walls during compaction and ejection by about 25 to 50% as compared to other lubricants, even if its content is much lower. In particular, it was shown that reducing the lubricant to a level of 0.25% could significantly improve the green density while keeping the ejection force at an acceptable level.

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