COMBINING ELECTROSTATIC DIE WALL LUBRICATION AND WARM COMPACTION TO ENHANCE GREEN AND SINTERED PROPERTIES OF P/M COMPONENTS

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ABSTRACT

The achievement of high density at reasonable cost would be a definite advantage for the production of P/M components requiring high static and dynamic properties. The use of warm compaction combined with the electrostatic die wall lubrication technique appears as a very attractive route to promote densification. Several studies carried out at a laboratory scale confirmed the potential of this processing route. However, the real benefits of combining both techniques on a production scale needed to be further explored. A study was conducted on an industrial mechanical press to determine to which extent the combination of both techniques can be useful in the achievement of higher density, and thus, higher mechanical properties. The benefits of this process in comparison with the conventional cold and warm pressing processes as well as the cold pressing process associated with the die wall lubrication technique were evaluated in this study and are presented in this paper.

INTRODUCTION

The production of P/M components with high static and dynamic properties for highly performance is increasingly required by the P/M industry. In particular, it is well known that increasing the sintered density of parts results in a significant improvement in static and dynamic properties. The final sintered density and mechanical strength of P/M parts are not only dictated by the powder formulation, but also by the compaction process and compacting conditions used, the part characteristics and the sintering behavior. The densification and ejection performance of powder mixes remains however one of the main factors to address when targeting very high density. The density level achieved when compacting metallic powders in a closed die is a function of the intrinsic ability of the powder mix to be densified, of the friction between the powder particles and the die walls, and of the springback of the compacted part after ejection [1].

Different processes are becoming increasingly available to the P/M industry to improve the densification and ejection performance of metallic powder mixes. The warm pressing process, which consists in pressing a preheated powder mix in a heated die, enables the fabrication of parts with high density and green strength by increasing the ductility of the ferrous powder particles [2, 3, 4]. The gain in density achieved by warm compaction versus cold compaction generally ranges
between 0.12 to 0.30 g/cm³. The density gain is usually larger for higher compacting pressure and less compressible powders [5, 6]. However, to take advantage of the beneficial effect of a moderate increase of the compacting temperature on densification, powder mixes must be properly designed, in particular the selection of the internal lubricant to provide adequate lubrication at die walls during both with regards to the compaction and ejection steps [5,7].

The die wall lubrication technique is also a promising avenue to promote green densities when high compacting pressures are used. This technique has been the object of several studies in recent years [8, 9, 10, 11, 12, 13]. The benefits of this technique consist in the possibility to significantly reduce the internal lubricant level in the powder mix, while maintaining good lubrication at die walls during the compaction and the ejection of parts. Additionally, this technique reduces the lubricant burn off in the sintering furnaces. In particular, when pressure is increased, the porosity in the compacted part tends to decrease until to the point where the volume of internal lubricant between metallic particles starts to inhibit the compaction. This is especially true with the highly compressible steel powder grades available nowadays, but also when warm compaction is used to improve the ductility of the metallic powders. Thus, the use of the die wall lubrication technique enables to postpone the occurrence of this inhibition of compaction and accordingly, favors higher green densities [14].

Although the die wall lubrication has been extensively studied at the laboratory scale, it is not widely used on a production scale because of the difficulty in controlling the amount of lubricant and especially the thickness and uniformity of the film deposited on die walls, and also because of the risk that improper die wall lubrication occurs sporadically causing accelerated die wear and even tooling seizure. Recently developed die wall lubrication systems have raised the performance and reliability of this technique [9,13,14] and are currently under evaluation by different P/M parts manufacturing companies.

The object of this paper is to further help identify the benefits, limitations and potential of a newly developed electrostatic die wall lubrication technique combined with the use of warm compaction to achieve high levels of sintered density and mechanical strength. The work was done on an industrial mechanical press by evaluating the compaction and ejection behavior, as well as the green and sintered properties, of short series of parts. The cold pressing process assisted with or without die wall lubrication and the current warm pressing process were also evaluated in a similar way and a complete comparison of these systems is presented.

**EXPERIMENTAL PROCEDURE**

Four different compaction processes were evaluated; cold pressing (CP), warm pressing (WP), cold pressing with die wall lubrication (DWL) and warm pressing with die wall lubrication (WPDWL). The material system used for this evaluation was ATOMET 4401, a water-atomized powder prealloyed with 0.85 % Mo and 0.15 % Mn, admixed with 4.0 % Ni, 1.5 % Cu and 0.6 % graphite. The reference mix (REF), which is a conventional non-binder treated mix containing 0.75 wt% of admixed Acrawax C (atomized) lubricant was used to evaluate the cold pressing process. The three other mixes i.e. the warm pressing mix (WP), the cold pressed die wall lubricated mix (DWL) and the warm pressed die wall lubricated mix (WPDWL) were binder treated. These mixes contained internal lubricant/binder systems specifically designed and suitable for the warm pressing and DWL processes. The WP mix contains 0.55 wt% of organic material while the DWL and WPDWL mixes contain only 0.25 wt% organic. Table 1 gives the composition and the pore free density (PFD) of the mixes used as well as their apparent density and flow rate measured in conformance with the MPIF standard 03 and 04 [15].
Table 1: Composition, apparent density, flow rate and theoretical density (PFD) of the FLN4-4405 modified mixes (93.9% ATOMET 4401, 4% nickel, 1.5% copper, 0.6% graphite) used to compare the different pressing processes.

<table>
<thead>
<tr>
<th>Identification of the mixes</th>
<th>Apparent density 25 °C (g/cm³)</th>
<th>Flow rate 25 °C (s/50 g)</th>
<th>Total organic material content (wt%)</th>
<th>PFD (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF</td>
<td>3.05</td>
<td>35.7</td>
<td>0.75</td>
<td>7.42</td>
</tr>
<tr>
<td>WP</td>
<td>3.18</td>
<td>30.0</td>
<td>0.55</td>
<td>7.51</td>
</tr>
<tr>
<td>DWL</td>
<td>3.36</td>
<td>25.2</td>
<td>0.25</td>
<td>7.67</td>
</tr>
<tr>
<td>WPDWL</td>
<td>3.20</td>
<td>32.3</td>
<td>0.25</td>
<td>7.67</td>
</tr>
</tbody>
</table>

The evaluation of the four materials/compaction processes was done by pressing 0.25 inch (6.35 mm) and 0.5 inch (12.7 mm) thick TR specimens with a 150 ton industrial mechanical press fully instrumented. The compaction rate was set at 5 parts/min. The TR specimen shape was used in order to enable comparison with previous work in the literature and allow the evaluation of the transverse rupture properties and therefore obtain a better judgement of the improvement given by every process at the green and sintered state, when pressing close to the highest reachable density. The testing conditions for the four compaction processes evaluated in this study are reported in Table 2. The sintering was done on a mesh belt laboratory furnace at 1120°C for 25 minutes in a 90/10% nitrogen/hydrogen atmosphere with a dew point below –40°C. The cooling rate was approximately 36°C/min between 650 and 400°C. The sintered samples were further submitted to a tempering step at 205°C for one hour in air.

Die wall lubrication was performed with the electrostatic die wall lubrication unit developed at IMI [14]. This system is based on the use of a confining block to favor a uniform deposition of lubricant on the die walls. The choice of the external lubricant to be used with this system was mainly dictated by the compaction temperature. Zinc stearate was used for the DWL process (cold compaction), while a proprietary die wall lubricant was used for the WPDWL process (warm compaction). All die wall lubrication parameters such as quantity sprayed, spray duration and gas pressure were optimized for each condition to visually reach the best uniformity in the die and have the lowest ejection forces on short pre-tests.

For each of the four materials/compaction processes evaluated in this study, the compacting conditions were first set up. Then, when part-to-part weight stability was reached (steady state), a series of 100 parts was produced uninterrupted. The following evaluations were made for each process:

- Part weights were recorded for all the parts compacted.
- Complete ejection curves were recorded for each condition and compaction process. Only representative ejection curves obtained at a compacting pressure of 60 tsi (827 MPa), will be presented in this paper to compare the ejection behavior related to each compaction process.
- The results of 5 equally spaced parts were used to determine the mean and standard deviation of the stripping ejection pressure, green density, springback or length expansion at ejection, green strength, and surface finish.
- For the sintered properties, the results of 5 other equally spaced parts were used to determine the mean and standard deviation of the transverse rupture strength (TRS standard #41 MPIF [15]) and the dimensional change from green and from die size.
Surface finish or roughness results were determined by using the Ra value (RMS of peak to peak values) along the long ejection sides of the TR specimens. The signal was acquired on a length of 0.5 inch (12.5 mm) with a roughness tester model Surftest 211 Mitutoyo on the scale 0.05 to 40 µm. Each reported result is the average of three consistent readings.

Table 2: Testing conditions of the different processes on a 150 ton Gasbarre industrial mechanical press (5 parts per minute).

<table>
<thead>
<tr>
<th>Identification : Compaction processes and related mixes</th>
<th>Binder/Lubricant (wt%)</th>
<th>Compacting temperature* (°C)</th>
<th>Die wall lubricant</th>
<th>Compacting pressure Tsi (MPa)</th>
<th>Part thickness inch (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF</td>
<td>0.75</td>
<td>50</td>
<td>No</td>
<td>40, 50, 60 (551, 684, 827)</td>
<td>0.25 (6.35)</td>
</tr>
<tr>
<td>WP</td>
<td>0.55</td>
<td>~125</td>
<td>No</td>
<td>40, 50, 60 (551, 684, 827)</td>
<td>0.25 (6.35)</td>
</tr>
<tr>
<td>DWL</td>
<td>0.25</td>
<td>50</td>
<td>ZnSt</td>
<td>40, 50, 60 (551, 684, 827)</td>
<td>0.25 (6.35)</td>
</tr>
<tr>
<td>WPDWL</td>
<td>0.25</td>
<td>~125</td>
<td>Proprietary</td>
<td>40, 50, 60 (551, 684, 827)</td>
<td>0.5 (12.7)</td>
</tr>
</tbody>
</table>

* Part temperature at the exit of the die

**RESULTS AND DISCUSSION**

**Green properties**

Figure 1a shows the green densities of the 0.25 and 0.5 inch thick TR specimens produced with the four compaction processes and mixes as a function of the compaction pressure. It is clear that at compaction pressure below 45 tsi, the warm compacted mixes (WP and WPDWL) reach higher green densities than the cold compacted mixes (REF and DWL) whatever the thickness of the TR specimens. It is also noteworthy that a compaction pressure below 45 tsi, the WPDWL mix containing only 0.25 wt% of binder-lubricant has similar compressibility to the WP mix containing a higher amount of binder-lubricant (0.55 wt%). This confirms that using die wall lubrication combined with warm compaction and a low amount of admixed lubricant allow to achieve quite elevated green densities at compacting pressure around 40 tsi [16, 17]. The behavior at lower compaction pressure was not evaluated in this study but it is believed that a higher lubricant content should be beneficial on density at compacting pressure lower than 40 tsi.

At high compaction pressure, a different behavior can be observed. Indeed, the density of the DWL and the WPDWL mixes continues to increase significantly as the compaction pressure increases up to 60 tsi, whereas the green densities of mixes compacted without die wall lubrication (REF and WP) either increases slowly (REF) or tend to reach a plateau at pressures close to 50 tsi (WP). As a result, the DWL mix leads to similar green densities to those obtained with the WP mix at 60 tsi. For example, with 0.25 inch thick TR specimens compacted at 60 tsi, green densities of 7.46 g/cm³, 7.35g/cm³, 7.38g/cm³ and 7.26 g/cm³ were obtained respectively for the WPDWL, WP, DWL and REF mixes.
The different densification behavior of the four mixes when the compaction pressure increases may be analyzed as a function of the percentage of the pore free density of the mixes, as shown in Figure 1b. In fact, the theoretical densification limit of a mix also called the pore free density (PFD) is a measure that takes into account the base metallic powder, but also the organic material and any other additive in a mix. This PFD can be significantly lower than the density of the metal-based powders depending on the amount of organic material added to the mix. In this study, the four mixes differ only by the amount of the binder-lubricant system in the mixes. The PFD was respectively 7.42 g/cc and 7.51 g/cc for mixes REF and WP and 7.67 g/cc for mixes DWL and WPDWL (see also Table 1). Besides, in practice, rather than reaching 100% of PFD values, simple compaction processes can reach no more than 98.5% of the PFD of a mix in the best case. The 98.5% limit is due to the part expansion after the relieving the compaction pressure and ejection from the die (springback).

Figure 1b shows that the REF and WP mixes, which tend to reach densification limit at high compaction pressures (50 and 60 tsi), have in fact high %PFD values between 97 and 98%. On the other hand, the significative increase of density as the compaction increases for the mixes containing a low amount of admixed lubricant and compacted with die wall lubrication (DWL and WPDWL) can be explained by their significantly lower %PFD. For example, at 50 tsi, the %PFD values are respectively 95% and 96.2% for the DWL and WPDWL mixes vs 97.2% and 97.5% for the REF and WP mixes. This clearly shows that the maximum practical relative density, which is about 98.5%, is reached at lower compacting pressure when lubricant content increases. This confirms the benefits of the die wall lubrication technique, which, by significantly reducing the internal lubricant level in the powder mix, enables to postpone the point where there is inhibition of compaction, and in this way can achieve higher densities.

![Figure 1](image_url)  
Figure 1: (a) Compressibility curves and (b) %PFD of ¼ and ½ inch thick TRS bars pressed at 5 strokes per minutes with the different compacting processes and mixes.
Expansion of green parts at ejection

Figure 2a shows the green length expansion of the 0.25 inch thick TR specimens as a function of the applied pressure and compaction process used. First, as expected, the springback always increases with the applied pressure whatever the compaction process used. On the other hand, it is clear that the lowest springback is observed for the parts produced by the WPDWL process. Indeed, lower springback values by nearly 40% at 40 tsi (551 MPa) and 30% at 60 tsi (827 MPa) are obtained as compared to the values obtained by the REF process. Moreover, parts compacted with the WP process undergo a large variation in springback at ejection as the compacting pressure is increased. Finally, for the cold compaction processes, slightly higher green expansion was observed for the DWL vs the REF process.

This higher green expansion for the cold DWL process compared to the reference might be due to a less complete particle rearrangement caused by the important decrease of the internal lubrication volume, even if this effect was not visible on the green density results.

At a compaction pressure of 40 tsi, the low springback observed for the two warm compaction processes (WP and WPDWL) may be explained by the higher ductility of the steel powders, which results in a larger amount of plastic deformation, metal to metal contact and cold welding between steel particles [3]. This likely contributes to reduce the effective stress in the part, and thus to reduce the green expansion.

At compaction pressures of 50 tsi or above, the larger springback obtained for the WP process vs the WPDWL process may be explained by the higher volume of binder-lubricant in the mix and the high values of %PFD reached. In fact, as the theoretical pore free density is approached, remaining air may be entrapped within porosity with the remaining organic material during pressing. This could result in high pressure within the parts. Thus, when the compacting pressure is relieved and the compact is ejected, the high internal pressure causes a significant expansion of the part, giving larger springback. This phenomenon could be even more important at temperature higher than 100°C due to the higher ductility of steel powders.

![Figure 2: Green length expansion at ejection for the different processes. (a) 0.25 inch thick TR specimens (b) 0.5 inch thick TR specimens.](image)
On the other hand, Figure 2b shows the green length expansion for the 0.5 inch thick TR specimens. Similar trends are observed vs the 0.25 inch thick TR specimens. However, the overall values are slightly higher, and particularly for the WP mix compacted at 60 tsi. This result is in line with studies carried out by St-Laurent and Chagnon who showed that the springback increased when increasing the part height [18].

**Green Strength**

The variation of green strength as a function of compaction pressure for the different processes used are shown in Figure 3. It can be seen that significantly higher green strengths are obtained when warm compaction combined or not with die wall lubrication is used. The level of green strength, (over 5000 psi, 34.5 MPa), is clearly sufficient for most green machining operations. For example, green strengths of 0.25 inch thick parts pressed at 60 tsi reach respectively 6506 and 6100 psi for mixes WP and WPDWL as compared to 1925 and 2070 psi for mixes REF and DWL. It is clear from Figure 3 that the compacting pressure, and thus, the density, has only a minor effect on green strength and is not a determining factor regarding green strength. Indeed, the different level of green strength, reported above, for parts produced by the DWL process (cold compaction) and the WP process, were obtained at similar densities, i.e. respectively, 7.38 g/cm$^3$ and 7.36 g/cm$^3$. In addition, the WP process has the advantage to give very high green strength at a relatively low compacting pressure. Green strength, not reported here, higher than 5000 psi (34.5 MPa) at only 30 tsi (414 MPa) has been reached with the WP mix.

The significantly higher green strengths obtained with the WP and WPDWL processes may be explained by two different phenomena. First, when warm pressing is used, the internal lubricant tends to move preferentially in the pores rather than between particles and the interlocking and microwelding between particles are enhanced [3]. Secondly, the polymeric components used in WP and WPDWL powder mixes have a significantly higher intrinsic mechanical strength than conventional lubricants [19]. When warm compaction is applied, these polymers have the ability to flow between particles and create strong and adhering polymeric bridges or network between the steel particles that strengthen the green specimens. The slightly higher green strength observed with the WP mix vs the WPDWL may be explained by the higher amount of polymeric component in the WP mix.

On the other hand, the low green strengths of parts produced by cold compaction (REF and DWL) can be explained by the nature of the internal lubricant in the powder mixes. Indeed, it is well known that lubricated mixes containing conventional lubricants lead to low green strength as compared to unlubricated mixes due to the formation of a film of low mechanical strength at the interface of metallic particles that limit their cohesion [3, 20]. It is noteworthy that the green strength of parts produced by the DWL process (cold compaction) remains low (2070 psi at 60 tsi) despite the high density obtained (7.38 g/cm$^3$) and the low level of internal lubricant.
However, higher green strength might have been obtained by these cold compaction processes (REF and DWL) if adequate polymeric binder-lubricant systems were used [19, 21].

**Ejection behavior**

The ejection pressure was recorded for each compacted part. Figure 4 shows typical ejection curves of 0.5 inch thick TR specimens compacted at 60 tsi (827 MPa) with DWL, WP and WPDWL mixes, and 0.25 inch thick TR specimens compacted from REF mix. These curves represent the variation of the ejection shearing stress, which corresponds to the ejection force required to eject the part out of the die, divided by the surface of the compact in contact with the die walls. The initial sharp increase of the shearing stress, called stripping pressure, is necessary to overcome the static friction at die walls, while the subsequent reduction of the ejection force is attributed to the lower dynamic friction coefficient and the asperity reduction at the surface of the compact resulting from friction wear. An interesting parameter, the ejection energy, was also evaluated from the area under the ejection curve. This parameter was used in this study rather than the sliding pressure, often cited in publications, which corresponds to the mean stress to move parts to the die entrance, since no “plateau” of the ejection shearing stress was observed for the type of parts and compacting conditions used in this study.

Figure 4 reveals that all the parts show a relatively good ejection behavior, whatever the compaction process used, with stripping pressures lower than 2 tsi (28 MPa) when a compacting pressure of 60 tsi (827 MPa) is used. However, among the different processes, the WPDWL process has the best ejection behavior with the lowest stripping pressure and ejection energy. Indeed, the area under the curves for the WP and the DWL processes are respectively 36% and 122% higher than for the WPDWL process. It should be mentioned here that the ejection performance is greatly dependent on the nature of either the internal or the die wall lubricant.

Figure 4: Ejection curves of TR specimens compacted at 60 tsi (827 MPa) with the four different processes: A) REF 0.25 inch thick, B) DWL, C) WPDWL, and D) WP, 0.5 inch thick.
Then, the values of ejection energy presented above indicate only the excellent behavior of the WPDWL process, but optimization of the DWL and WP processes would improve the ejection performance of the mixes compacted with these processes.

Figures 5 and 6 report more specifically the stripping pressures measured as a function of the compacting pressure for the different processes and parts tested in this study, as well as the standard deviation of these values calculated from series of 100 parts for each compacting conditions.

All processes show acceptable ejection forces at compacting pressures up to 60 tsi (827 MPa). The highest stripping pressures were obtained for the ejection of 0.5 inch thick TR specimens compacted with the WP process, but however remained relatively low with values below 2 tsi (28 MPa). On the other hand, the influence of the part thickness on the stripping pressure is not the same for all the processes. Indeed, the stripping pressure at a given compacting pressure increases significantly for the WP process when the part thickness is doubled (respectively 13, 34 and 31 % at 40, 50 and 60 tsi), while for the cold DWL process, the stripping pressure increases more moderately (11, 22 and 18 %). For the WPDWL process, no negative influence of the part thickness except at 40 tsi was observed. The higher influence of the part thickness on the stripping pressures for the WP process suggests that it could be the first process to show a lack of lubrication at the die wall for a more demanding part having a higher surface in contact with the die walls.

On the other hand, it is noteworthy that, besides being insensitive to the part thickness, the WPDWL process shows the lowest stripping pressure at the highest densities reached in this study and even shows a decrease of its stripping pressure as the compacting pressure is increased. As compared to the cold DWL process, this result may be partly explained by the highest %PFD reached that could cause, at high pressure, the squeeze out of a larger quantity of internal lubricant towards the die walls, thus improving the ejection performance.

Furthermore, standard deviations of the stripping pressures presented in Figure 6 indicate that the two most stable processes regarding ejection pressures are those using the die wall lubrication technique (DWL and WPDWL). These results confirm the stability and reliability of the electrostatic die wall lubrication technique used in this study [14].

![Figure 5: Stripping pressure of the 0.25 and 0.5 inch thick bars pressed with the different processes.](image)

![Figure 6: Standard deviation of the stripping pressure for each series of 100 parts produced.](image)
Finally, to better discriminate the ejection performance of the different compaction processes and to better identify their limitations, it would be interesting, in a future work to evaluate a more demanding part with a higher aspect ratio (sliding surface/cross section) than 0.25 and 0.5 thick TR specimens.

**Surface Finish**

The surface finish of green compacts was evaluated by measuring their surface roughness. Figure 7 presents the roughness Ra values measured as a function of the compacting pressure for the different compaction processes and thicknesses of the bars. Similar results were obtained whatever the compaction process used. Indeed, no significant difference was observed, considering the limits in the precision of the measuring technique.

It is worth mentioning here that if the die wall lubrication unit is not well configured or if the spray parameters are not well adjusted, parts having a bad surface finish may be obtained. Indeed, regarding the die wall lubrication unit used in this study, and described in a previous study [14], it is important to mention that the confining block design and the spray parameters were selected carefully in order to achieve a uniform coating on the tooling. Indeed, any turbulence pattern should be eliminated to avoid non-lubricated area that could cause galling of the tooling or lubricant accumulation particularly in corners that could cause some voids in the parts.

Furthermore, it is important to know that every lubricant used requires its proper spraying conditions to eliminate electrostatic agglomeration susceptible of affecting the surface roughness of the parts.

![Figure 7: Roughness measurement of the 0.25 and 0.5 inch thick green TR specimens pressed with the different processes.](image-url)
Sintered properties

Density and Transverse rupture strength

Figure 8 shows the densities and the transverse rupture strengths of the sintered and tempered 0.25 inch thick TR specimens produced by the four compaction processes, as a function of the compacting pressure. Looking to the sintered densities, similar trends were obtained as compared to the green densities. Indeed, for a compacting pressure of 60 tsi, high sintered densities of 7.48 g/cm³, 7.44 g/cm³, 7.40 g/cm³ and 7.29 g/cm³ were obtained respectively for the WPDWL, DWL, WP and REF processes.

It is however important to note that higher densification was seen for the parts produced by the cold DWL process versus the other processes. Indeed, for this process, a gain in density of 0.09, 0.08 and 0.07 g/cm³ was respectively observed for compacting pressures of 40, 50 and 60 tsi, compared to only 0.05, 0.01 and 0.02 g/cm³ for the REF process, 0.01, 0.03 and 0.03 g/cm³ for the WPDWL process and 0.01, 0.03 and 0.04 g/cm³ for the WP process. This higher densification during sintering results, in Figure 8a, in crossover points between the DWL and the WP curves at significantly lower compacting pressures than in the compressibility curves in Figure 1a. (~52 tsi vs. 57 tsi). The compacting pressure needed to achieve higher density with the DWL process than with the REF process is also lowered compared to the compressibility curves. The densification of a part during sintering is related to two phenomena: the volume change due to diffusion and microstructural change which is affected by the green density, and the weight loss due to organic burn-off and reduction of oxygen. In this case, the higher densification obtained was mainly related to the lower amount of internal lubricant content in mixes DWL and WPDWL, which gave lower weight loss [5]. In the case of the WPDWL mix however, the gain in density was lower due to the higher green densities achieved which limit further densification.

Figure 8b shows the sintered strength as a function of the compacting pressure. Typically, very elevated sintered strengths in the range 280 to 320 ksi (1.9 to 2.2 Gpa) were achieved for mixes DWL, WPDWL and WP. Slightly lower sintered strengths were achieved with REF mix due to the lower density achieved as shown in Fig. 8a. Sintered strengths in the range of 200-220 ksi (1.4-1.5 GPa) are usually obtained at 6.8 g/cm³ with FLN4-4405 + 1.5 % Cu material compacted at room temperature. It can also be seen in Fig 8b that the TRS remained quite stable with the compacting pressure for the high compacting processes. Normally, a gain in TRS is obtained when increasing the density, and thus the compacting pressure. Indeed, a sintered strength of about 340 ksi was reached at a density of 7.40 g/cm³ with WPDWL and DWL pressed on a hydraulic laboratory press and sintered and tempered under the same conditions as those used in this study. It should be noted the microstructure formed for such a formulation is very heterogeneous and is constituted mainly of areas of bainite, martensite and Ni-rich retained austenite. The proportion of martensite is significant in such materials as shown in ref [22]. It is known that large scatter in transverse rupture strength is obtained when the proportion of martensite is high. Further studies will be done to better understand the trend observed in Figure 8b.
Dimensional change

The influence of the compacting pressure on the dimensional change of the four materials/processes during sintering is shown in figure 9. The analysis of the dimensional change during sintering must be put in relation with the volume expansion of the green parts at ejection from the pressing die to be easily understood.

First, for all the materials, dimensional change from die size becomes more positive as the compacting pressure, and thus the density increases, Fig. 9a. In fact, typically, dimensional change versus die follows the same trend as the springback. It was already shown that increasing the compacting pressure led to an increase in springback.

The densification of a part during sintering is better illustrated by the dimensional change versus green size. Indeed, a positive dimensional change versus green size indicates that part grow during sintering while a negative dimensional change shows the opposite. Dimensional change versus green size as a function of compacting pressure is given in Figure 9b. It can be seen that all the parts shrank during sintering. Less dense parts usually gives higher shrinkage during sintering. The variation in dimensional change is also less important than that obtained versus die size as the compacting pressure increased.

Furthermore, it is interesting to note for mix WP that the dimensional change becomes more negative as the compacting pressure increases contrary to what is observed with the other processes and with the dimensional change versus die size. This change in behavior is not fully understood but springback has likely played a role. Indeed, WP mix shows the highest increase in springback when compacting pressure was increased. It was already shown in a previous study that an increase in springback usually gives higher shrinkage during sintering [18].
CONCLUSION

This study was undertaken to evaluate the benefits, limitations and potential of the warm pressing process combined with the die wall lubrication technique (WPDWL) in comparison with the conventional cold pressing (REF) and warm pressing (WP) processes, and the cold pressing process combined with the die wall lubrication technique (DWL). The most relevant conclusions that can be drawn are as follow:

- The electrostatic die wall lubrication system used in this study, which is based on the use of a confining block to facilitate the lubricant deposition on the die walls, allowed the production of parts on an industrial mechanical press with a good stability at a production rate of 5 parts/min. Good surface finish with lower ejection forces were obtained with the die wall lubrication technique as compared to the other compaction processes studied at compacting pressure ranging between 40 and 60 tsi.
- The combination of the warm compaction and electrostatic die wall lubrication techniques allows achieving the highest density while giving the lowest stripping pressure and ejection energy in this study. Green density in excess of 7.4 g/cm³ was obtained at 55 tsi or above.
- The use of die wall lubrication technique combined with a reduction of the internal lubricant content allowed to obtain a continuously increase in density by increasing the compacting pressure from 40 to 60 tsi. The density leveled off at about 45 tsi with the warm compaction technique due to the higher amount of internal lubricant.
- The warm compaction processes combined or not with the die wall lubrication technique allowed to reach green strength above 5000 psi (34.5 MPa) for compacting pressure above 40 tsi (551 MPa).
- Transverse rupture strength above 300 000 psi (2050 MPa) was achieved in this studies due to high sintered densities achieved.
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