PERFORMANCE OF MIXES WITH NEW LUBRICANT SYSTEM BASED ON CHROMIUM ALLOYED POWDERS

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ABSTRACT

Chromium pre-alloyed powders are used in an increasing number of PM applications. These pre-alloyed Cr-powders are attractive as they combine high mechanical strength with cost effectiveness. However, due to the solution hardening effect of the pre-alloying elements, mixes based on pre-alloyed powders obtain lower compressibility compared to mixes based on pure iron powders. Therefore, Cr alloyed powders can be more demanding to compact and require more efficient lubricants.

The search for higher performance PM components calls for higher density of the sintered materials. To achieve this, a lower amount of lubricant is a prerequisite. To obtain sufficient lubrication, also at a lower amount of lubricant, heating of the tool die is an attractive approach.

A new, highly efficient lubricant system for demanding applications has been developed. In this paper this new lubricant system is compared to amide wax as lubricant in mixes based on chromium alloyed powders. Data of the performance of the different mixes during compaction of components under production conditions is presented. Improved mechanical properties from the increase in density are also reported.

INTRODUCTION

In the PM industry copper, nickel and molybdenum are the most widely used alloying elements, whereas chromium and manganese are the more common alloying elements in wrought steels. Copper, nickel and molybdenum have the advantage of low affinity to oxygen, which is beneficial in the sintering process of PM steels. A drawback with using nickel and molybdenum as alloying elements is the relatively high cost for these elements. Chromium and manganese are generally more cost efficient alloying elements in steels. By using N_2/H_2 based sintering atmosphere with low partial pressure of oxygen sintering of chromium alloyed sinter steels is feasible[1]. By having chromium pre-alloyed instead of added as a master alloy

makes the material less sensitive to oxidation due to lower activity of the alloying element when pre-alloyed.

The hardenability of chromium alloyed sintered steels makes them well suited for sinter hardening[2] and mechanical properties are further enhanced if high temperature sintering is utilized[3]. However, due to the solution hardening effect of the pre-alloying elements, mixes based on pre-alloyed powders have lower compressibility compared to mixes based on pure iron powders. Therefore, Cr alloyed powders can be more demanding to compact and efficient lubricants are needed.

High density is generally desirable for high strength sintered parts. By using a highly efficient lubricant in lower amount than customary, the compressibility of the powder mix can be increased. To further enhance the lubrication, and thereby allow a lower amount of lubricant to be used, heated tool die is an attractive method[4].

In this paper two grades of pre-alloyed Chromium steels were investigated, these were the standardized grades FL-5208 with 1.5% Cr and 0.2% Mo and FL-5305 with 3.0% Cr and 0.5% Mo (standard designations according to MPIF standard 35). Two mixes were made of each alloying system. The high performance Intralube® E mix was benchmarked with a Premix with amidewax as lubricant. Amidewax, ethylene-bis-stearamide, is one of the most widely used lubricants in the PM industry today.

EXPERIMENTAL

This paper presents four evaluations of the two types of mixes, covering different aspects of the performance of the mixes. Compressibility and ejection properties were measured by using an instrumented hydraulic press. Press campaigns carried out in a production press with a multi-level tooling demonstrated the performance under production conditions. Besides the compressibility and ejection force, this evaluation also included weight control of the parts. The lubrication performance was stressed by compaction of bushings, of which the height was incrementally increased until surface quality deteriorated and scoring was seen on the surface of the parts. Finally, sintered properties were compared of the two types of mixes in both alloying systems.

First investigation: Compressibility and ejection performance

For the first, second and fourth investigation four mixes were made, see Table 1 for mix type, compositions and designations. Astaloy CrL is a pre-alloyed steel powder with 1.5% Cr and 0.2% Mo and Astaloy CrM is a steel powder pre-alloyed with 3.0% Cr and 0.5% Mo. The Intralube® E mixes had 0.2% and 0.15% lower lubricant amount respectively, compared to the corresponding Premix. The batch size of each of the mixes was 1 ton.

Table 1: Mixes

Designation	Type of mix	Composition
FL-5208 P	Premix	Astaloy CrL + 1% Cu + 0.65% Gr. + 0.3% MnS + 0.8% Amidewax
FL-5208 I	Intralube® E mix	Astaloy CrL + 1% Cu + 0.65% Gr. + 0.3% MnS + 0.6% Lube E
FL-5305 P	Premix	Astaloy CrM + 0.55% Graphite + 0.8% Amidewax
FL-5305 I	Intralube® E mix	Astaloy CrM + 0.55% Graphite + 0.65% Lube E

Flow and apparent density were measured according to ISO 4490 and ISO 3923-1 respectively and the graphite and lubricant contents of the mixes were analysed. Of each mix ring shaped parts were pressed at 500 MPa (36 Tsi), 650 MPa (47 Tsi) and 800 (58 Tsi). The dimensions of the rings were Ø55/45 mm (2.17/1.77 inch) and height 15 mm (0.59 inch). The press used in this investigation was an instrumented 125 ton hydraulic press, Result EHP-125. During ejection the positions and forces were logged and the

static ejection force (stripping force) was measured as well as the energy to eject the parts. The ejection energy was calculated by integration of the force-displacement curve from the ejection. Both the ejection force and energy were expressed in relation to the envelope surface of the rings.

Compaction of Premix was carried out with the tool die at room temperature while Intralube® E mixes were compacted with the tool die heated to 70°C (158°F).

Second investigation: Continuous compaction of multi-level parts

In the second investigation multi-level parts were compacted in a Dorst TPA250/3HP hydraulic press. The press tool had four upper punches and three lower punches. The shape of the parts compacted is illustrated in Figure 1. The outer diameter of the parts was 77 mm (3,0 inch) and the height of the outer segment was 21 mm (0.83 inch). The weight of the parts was ~470g, exact weight depending on the density level. The press rate was 6.3 parts/minute.

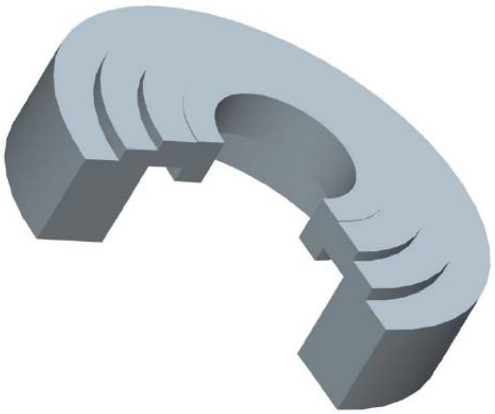


Figure 1: Drawing of the multi-level part (a slice is cut out to show the cross section)

For each mix, two compaction runs were made at a compacting pressure of 440 MPa (32 tsi) and 560 MPa (40 tsi) respectively. The maximum compacting pressure was limited to 560 MPa (40 tsi) since the capacity of the press, 2500 kN, was reached. As in the first investigation the tool die was not heated when Premix parts were compacted while the tool die was heated to 70°C (158°F) when Intralube® E mixes were compacted. Each pressed part was automatically weighed and the parts were sampled directly after the ejection to measure the temperature of the outer surface.

Pressed parts were sampled for documentation of the surface finish and measurement of green density by weighing in air and water.

Third investigation: Max height of bushings

In the third investigation the same two types of mixes, Premix and Intralube® E mix, based on Astaloy CrM were compared. In this case the amount of lubricant was the same in both mixes. The batch size of the mixes was 500 kg.

Table 2: Mixes

Designation	Type of mix	Composition
FL-5305 P-2	Premix	Astaloy CrM + 0.5% Graphite + 0.6% Amidewax
FL-5305 I-2	Intralube® E mix	Astaloy CrM + 0.5% Graphite + 0.6% Lube E

The dimensions of the bushings were Ø40/20 mm (1.57/0.79 inch) and height was varied in the range 25 – 50 mm (0.98 – 1.97 inch). A 100 ton hydraulic Gasbarre press was used for the trials. The material in the

tool die was carbide and the press tool had a floating core rod. The tool die was heated to 60°C (140°F) in the trials with both lubricants.
The evaluation of FL-5305 P-2 was started with a height of 25 mm (0.98 inch) and 30 parts were pressed, thereafter the height was increased in increments of 2.5 mm (0.1 inch) and 30 parts of each height were pressed. This procedure was repeated until the height was reached where scratches appeared on the surface of the parts, which was an indication of insufficient lubrication. The tallest height with scratch free surface was defined as the max height that could be achieved for the mix and part geometry. Max height test was carried out at two compacting pressures, 600 MPa (43 tsi) and 800 MPa (58 tsi).

In the trials green density, temperature of the parts after ejection and the surface quality were evaluated.

Fourth investigation: Sintered properties
The same four mixes as in the first and second investigation were also used in this investigation. Of each mix tensile test parts according to ISO 2740 and impact energy bars according to ISO 5754 were compacted. The specimens were compacted at three compacting pressures, 500 MPa (36 tsi), 650 MPa (46.8 tsi) and 800 MPa (57.6 tsi). The specimens of Premix were compacted without heating the tool die while the specimens of Intralube® E mixes were compacted with the tool die heated to 70°C (158°F).

The green and sintered densities of the tensile test bars were measured by weighing in air and water. Dimensional change from green to sintered specimen was measured. Hardness, HV10, tensile properties and impact energy were measured. The copper and carbon contents were analysed for the tensile test bars.

RESULT AND DISCUSSION

Analyses of the mixes
The analyses of the mixes used in the first and second investigations are presented in Table 3. Flow and apparent density were similar for Premix and Intralube® E mix. The apparent density was slightly higher for Premix while Flow was faster for Intralube® E mix in the case of FL-5208.

Table 3: Analyses of the mixes

Designation	Type of mix	AD [g/cm³]	Flow [s/50g]	Graphite [%]	Lubricant [%]
FL-5208 P	Premix	2.96	33,8	0,65	0,80
FL-5208 I	Intralube® E mix	2.91	31,8	0,68	0,62
FL-5305 P	Premix	2,83	34,3	0,58	0,83
FL-5305 I	Intralube® E mix	2,80	34,2	0,59	0,66

First investigation: Compressibility and ejection performance
Compressibility curves from the first investigation are presented in Figure 2. As a consequence of the higher alloying content in FL-5305 compressibility was somewhat lower compared to FL-5208. For both alloying systems the green density was increased by ~0.1 g/cm³ by using Intralube® E mix in combination with heated tool die. With Intralube® E mix similar or higher density could be achieved at 600 MPa (43 tsi) as with the Premix at 800 MPa (58 tsi). A lower compaction pressure can be important for components with narrow sections requiring press tools with slender punches. The higher compressibility was due to a combination of the more efficient lubrication, the lower amount of lubricant and the heated tool die.

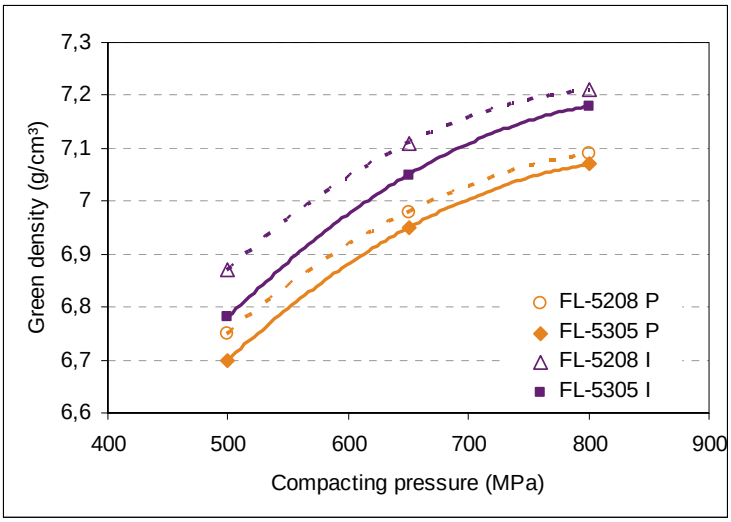


Figure 2: Compressibility curves

In Figure 3 ejection energy is plotted versus the green density. FL-5208 I and FL-5305 I were significantly lower in ejection energy compared to the corresponding Premix. The lower ejection energies were achieved even though the lubricant contents were 0.2% and 0.15% lower, respectively. An influence from the alloying content of the base powders can also be seen, the ejection energies of FL-5305 were higher compared to FL-5208. This effect was seen on both types of mixes.

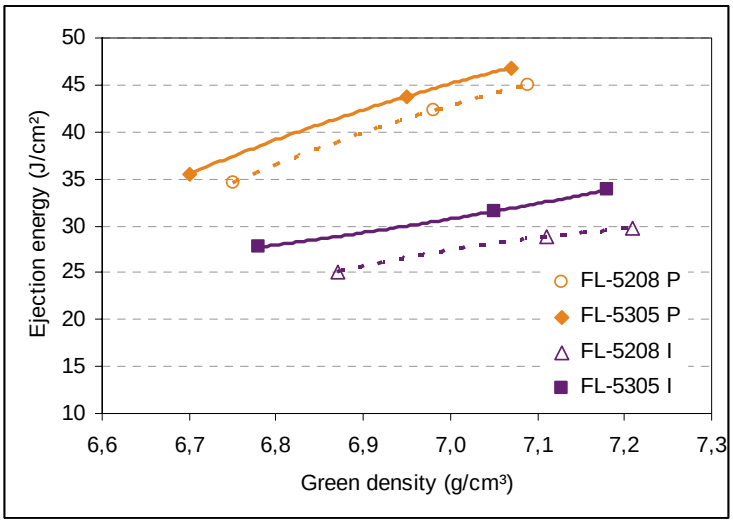


Figure 3: Ejection energy versus green density

There was less difference between the two types of mixes in terms of ejection force, see Figure 4. In the case of FL-5208 I the ejection force was lower compared to FL-5208 P when comparing at the same density level. For FL-5305 ejection force was similar at 7.06 g/cm³ but at lower density level the ejection force was lower with FL-5305 P.

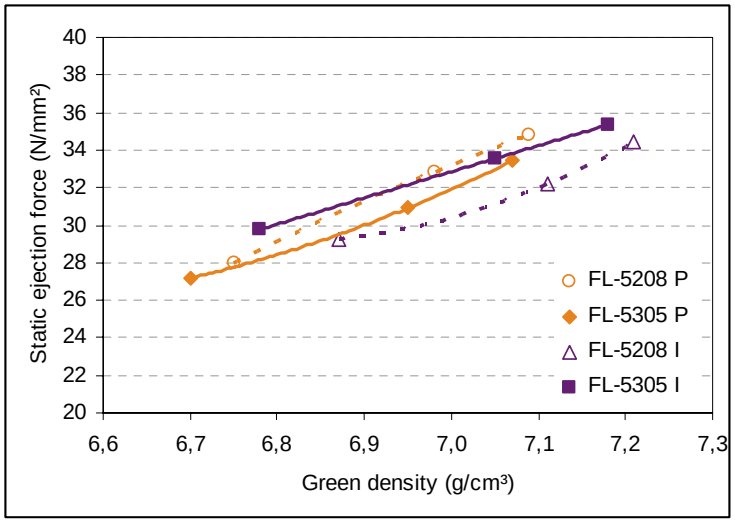


Figure 4: Ejection force versus green density

Second investigation: Continuous compaction of multi-level parts

In Table 4 the results of the continuous compaction trial are summarized. All parts had shiny surfaces without any scoring or other signs of insufficient lubrication.

Table 4: Results from continuous compaction

	Comp. Pressure Level	Mean [MPa] (tsi)	Ejection force [kN]	Weight Mean [g]	Std.dev [g]	Std.dev [%]	Part Temp. [°C] (°F)	Green density [g/cm³]	Sintered density [g/cm³]
FL-5208 P	Low	437 (31,5)	180	465,4	1,15	0,25%	52 (126)	6,72	6,70
	High	550 (39,6)	217	484,8	1,13	0,23%	59 (138)	6,92	6,90
FL-5208 I	Low	434 (31,2)	175	463,9	1,14	0,25%	62 (144)	6,72	6,71
	High	548 (39,5)	183	483,1	1,06	0,22%	67 (153)	6,95	6,93
FL-5305 P	Low	433 (31,2)	180	452,5	0,77	0,17%	56 (133)	6,60	6,58
	High	562 (40,5)	238	477,9	0,84	0,18%	62 (144)	6,89	6,88
FL-5305 I	Low	444 (32,0)	175	457,5	0,89	0,19%	63 (145)	6,66	6,67
	High	557 (40,1)	192	482,9	0,92	0,19%	68 (154)	6,93	6,91

In Figure 5 the maximum ejection force of the multi-level parts is presented. At the lower compacting pressure the ejection force was very similar for all four mixes. At the higher compacting pressure the ejection force of FL-5208 I and FL-5305 I were significantly lower compared to the corresponding Premix.

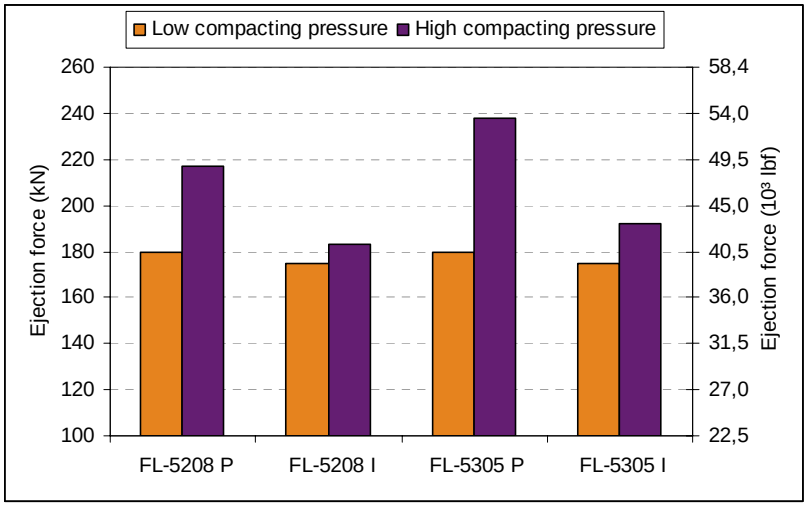


Figure 5: Ejection force of multi-level parts

The temperature of the parts after ejection is presented in Figure 6. With higher compacting pressure the temperature of the parts after ejection was higher. The temperature of the parts made of FL-5208 I and FL-5305 I was, as can be expected, higher due to the heating of the tool die. The temperature difference between parts made of the mixes pressed with heated tool die and the corresponding Premix was in the range 6°C (11°F) and 10°C (18°F).

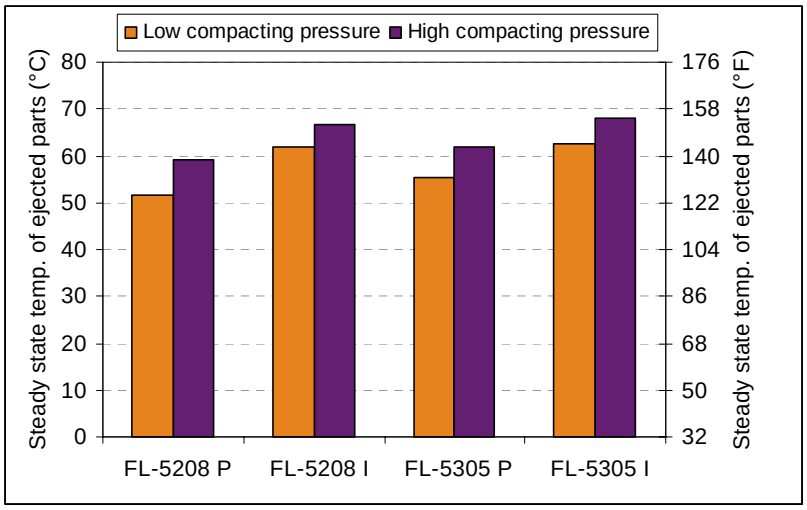


Figure 6: Temperature of multi-level parts after ejection

The weight scatter of the pressed parts is presented in Figure 7. The only significant difference was that the FL-5305 mixes scattered less than the FL-5208 mixes. There was no significant difference in weight scatter of the pressed parts between the two types of mixes.

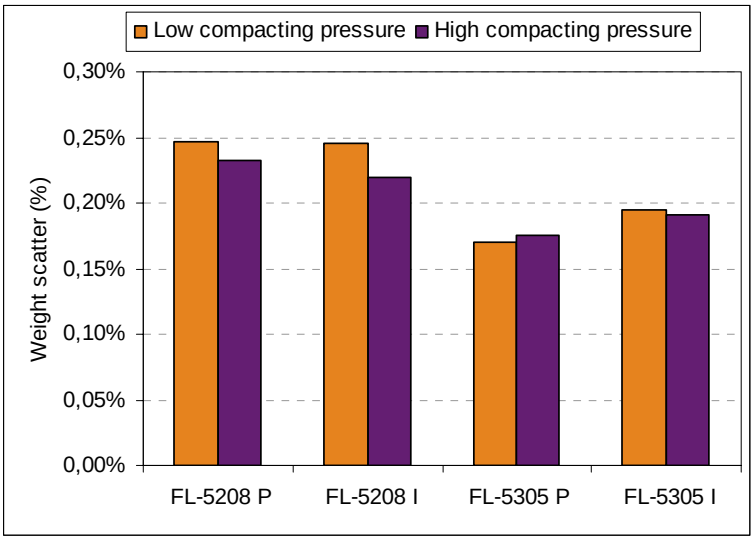


Figure 7: Weight scatter of multi-level parts expressed as one standard deviation

In Figure 8 the green density of the parts is presented. In general the Intralube E mixes were somewhat higher in green density. To benefit from the lower lubricant content of the Intralube E mixes, higher compacting pressure is required. The impact of the heating of the tool die was smaller compared to what was found in the first investigation. This can be explained by the size of the parts, with the thin walled ring in the first investigation a larger volume of the part was influenced by the heating than in the case of the larger part used in this investigation. The heat softens the lubricant which improves the compressibility as well as improving the lubrication.

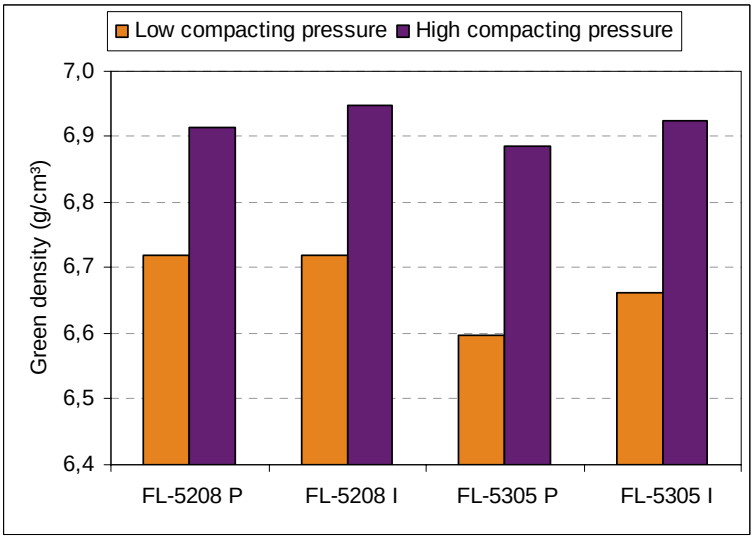


Figure 8: Green density of multi-level parts

In Figure 9 the sintered density of the parts is presented. The sintered densities were quite similar to the green densities and the relation between the two types of mixes was similar.

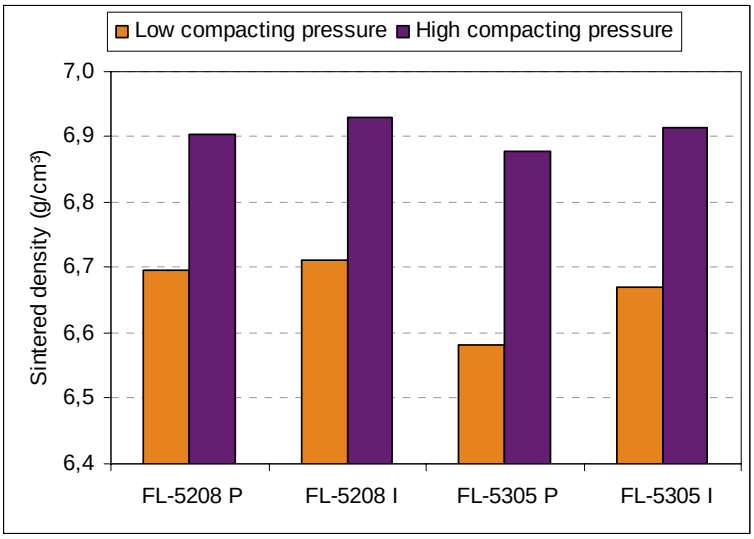


Figure 9: Sintered density of multi-level parts

In this second evaluation it was demonstrated that the Intralube E mixes were working well for compaction of this multi-level part. Despite the lower amount of lubricant compared to Premix, ejection force was lower and the compressibility slightly higher. However, the full potential of increased compressibility was not explored in this investigation due to the limitation in compaction pressure. The weight stability showed that the Intralube E mixes were comparable to Premix with amidewax as lubricant regarding filling of the tool die.

Third investigation: Max height of bushings

In this third investigation the experimental design differed from the two previous investigations, the two mixes FL-5305 I-2 and FL-5305 P-2 had the same lubricant content, 0.6%, and the tool die was heated to 60°C (140°F) during the compaction of both mixes.

In Figure 10 the height of the highest bushings with scratch free surface which were pressed of the two mixes at 600 MPa (43 tsi) and 800 MPa (58 tsi) are presented. At 600 MPa (43 tsi) 20 mm (0.79 inch) higher bushings could be compacted with FL-5305 I-2 compared to FL-5305 P-2. At 800 MPa (58 tsi) the difference in achievable height with the two mixes was 12.5 mm (0.49 inch).

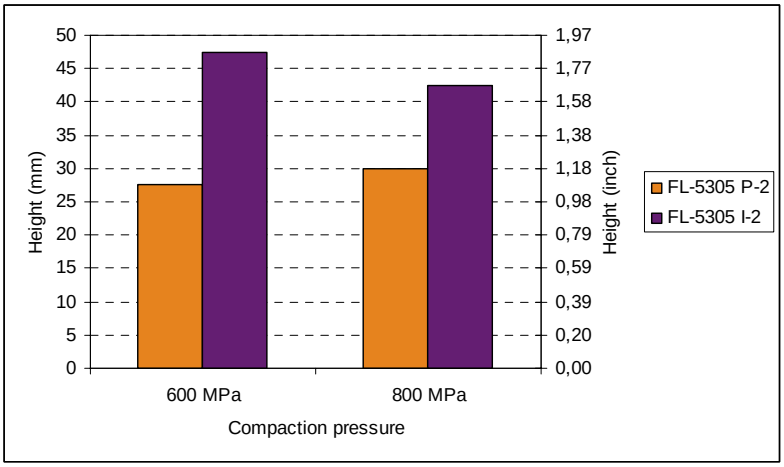


Figure 10: Highest scratch-free bushings

In Figure 11 the part temperature after ejection is plotted versus the height of the compacted parts. As expected the temperature of the parts after ejection increases with height of the parts since more heat is generated from the ejection. Comparing the temperature of the parts of the two mixes with the same height the temperature was higher for FL-5305 P-2 than FL-5305 I-2, which indicates better efficiency of the lubricant of FL-5305 I-2. The temperature increased more for FL-5305 P-2 than for FL-5305 I-2 when the compacting pressure was increased from 600 MPa (43 tsi) to 800 MPa (58 tsi).

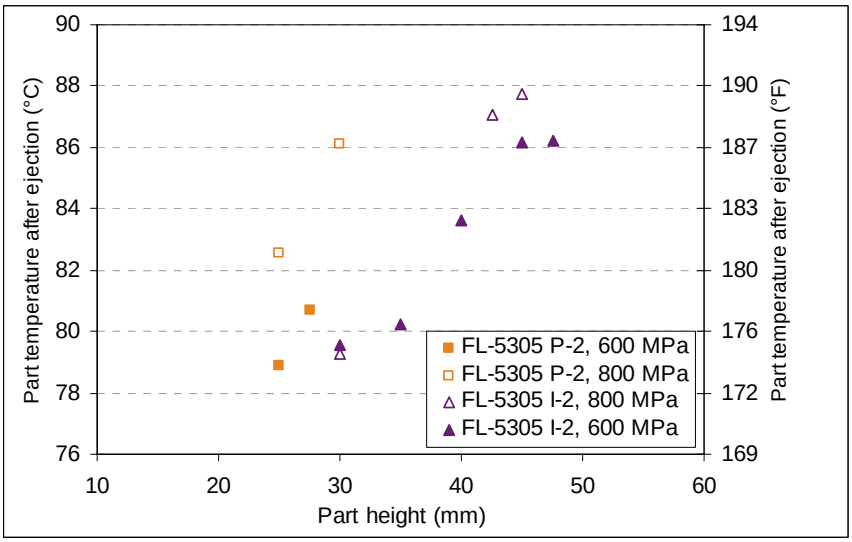


Figure 11: Part temperature after ejection

Fourth investigation - Sintered properties

In Table 5 sintered properties of the four mixes, each compacted at three compaction pressures, are presented.

Table 5: Mechanical properties

Designation	Comp. pressure [MPa] (tsi)	Dim. change [%]	Sint. density [g/cm³]	Mechanical properties HV10	Rm [MPa] (10^3psi)	$Rp_{0,2}$ [MPa] (10^3psi)	E [GPa] (10^6psi)	A [%]	IE [J] (Ft-lb)	Chemical analyses C [%]	Cu [%]
FL-5208 P	500 (36)	-0,05	6,80	191	604 (87,6)	495 (71,7)	121 (17,5)	0,6	11,3 (8,3)	0,65	1,01
	650 (47)	-0,04	6,99	217	679 (98,4)	543 (78,8)	139 (20,1)	0,7	15,7 (11,6)	0,66	1,02
	800 (58)	-0,04	7,09	245	749 (109)	582 (84,4)	144 (20,9)	1,0	16,4 (12,1)	0,66	1,05
FL-5208 I	500 (36)	0,00	6,88	203	667 (118)	519 (84,1)	130 (17,7)	1,0	12,3 (10,2)	0,65	1,06
	650 (47)	0,03	7,08	225	722 (130)	558 (93,4)	140 (20,0)	1,1	16,8 (14,3)	0,64	1,07
	800 (58)	0,03	7,17	248	780 (144)	598 (99,4)	148 (21,9)	1,2	20,1 (17,1)	0,65	1,07
FL-5305 P	500 (36)	-0,15	6,76	251	813 (96,7)	580 (75,3)	122 (18,9)	0,9	13,8 (9,1)	0,52	0,02
	650 (47)	-0,15	6,99	274	896 (105)	644 (80,9)	138 (20,2)	1,0	19,4 (12,4)	0,51	0,02
	800 (58)	-0,17	7,10	309	990 (113)	686 (86,7)	151 (21,5)	1,2	23,2 (14,8)	0,52	0,02
FL-5305 I	500 (36)	-0,11	6,83	264	843 (122)	602 (87,3)	127 (18,4)	1,0	15,2 (11,8)	0,51	0,02
	650 (47)	-0,07	7,08	302	978 (142)	685 (99,3)	146 (21,2)	1,2	21,9 (16,1)	0,51	0,02
	800 (58)	-0,07	7,19	334	1077 (156)	733 (106)	153 (22,2)	1,2	24,2 (17,8)	0,51	0,02

In Figure 12 the sintered density of the tensile specimens is plotted versus the compacting pressure. The increase in compressibility that was achieved with the Intralube® E mix and heated die compaction was maintained also after sintering and the sintered density was 0.07 – 0.09 g/cm³ higher compared to the Premix.

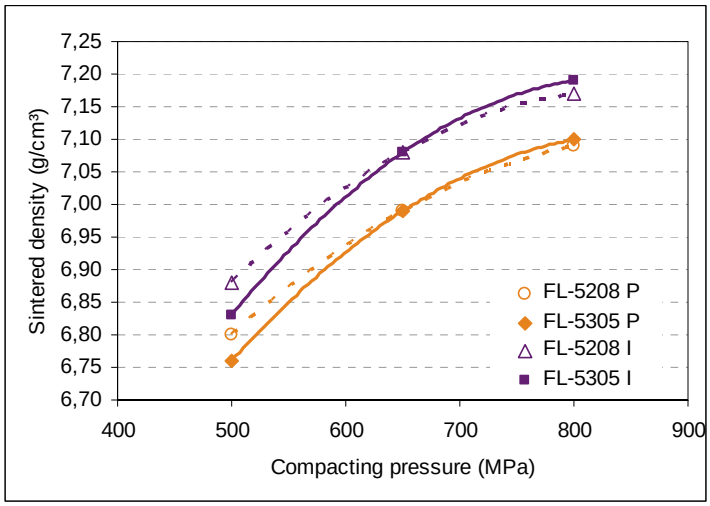


Figure 12: Sintered density of tensile specimens

By using Intralube® E mix and warm die compaction the mechanical properties increased proportional to the increase in sintered density. In Figure 13 the tensile strength is plotted versus the compacting pressure. Materials based on Intralube® E mix were 4 – 10% higher in tensile strength compared to the same material based on a Premix, comparing the properties achieved at the same compacting pressure.

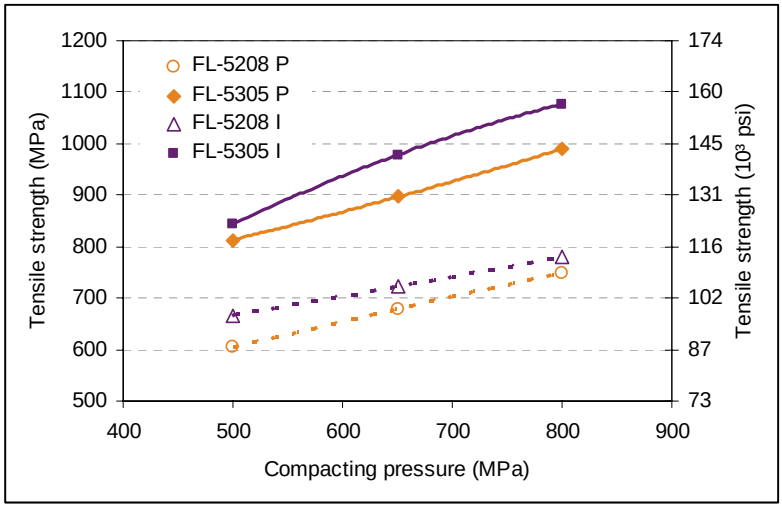


Figure 13: Tensile strength versus compacting pressure

Ductility of the materials increased even more by the use of Intralube® E mix , see Figure 14 where impact energy is plotted versus compacting pressure. Impact energy increased by 4 – 23%

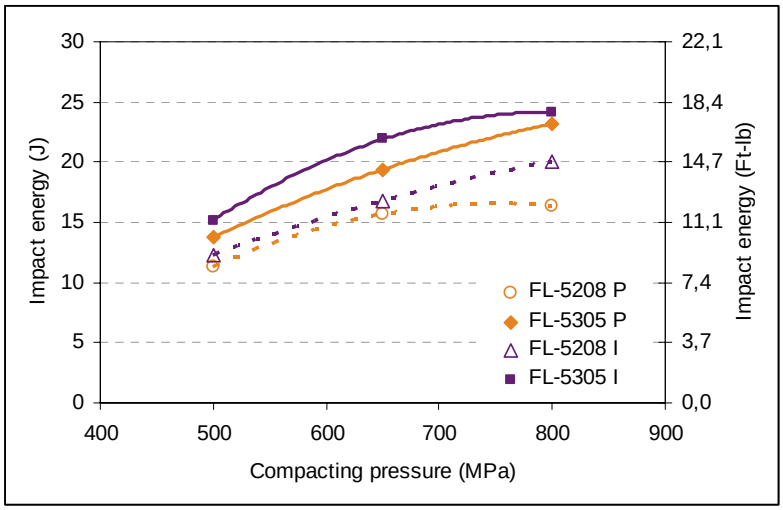


Figure 14: Impact energy versus compacting pressure

CONCLUSIONS

Based on the results where Intralube® E mixes based on chromium alloyed base powder were compared to Premix of the same nominal compositions and with amidewax as lubricant, it has been demonstrated that:

- Flow and apparent density are comparable between Intralube® E mix and Premix with amidewax as lubricant.
- Intralube® E mix is very efficient in terms of lubrication. In combination with heated tool die the lubricant content was decreased by 0.15 – 0.2% and still provided better lubrication than the reference.
- With decreased lubricant content and heated tool die the compressibility is improved, this is most significant at higher compacting pressure.
- The improved lubrication of Intralube® E mix allows significantly higher parts to be compacted with scratch free surface of the parts after ejection.
- Weight consistency is comparable between the Intralube® E mix and Premix.
- Mechanical properties, strength as well as ductility, increased proportionally with the increase in sintered density which was achieved by Intralube® E mix in combination with warm die compaction.

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