High Performance Mixes with New Lubricant System

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Abstract
To improve the performance of PM components higher density is one important approach. Lower amount of lubricant is often required to increase the compressibility of a mix. To obtain sufficient lubrication also at lower amount of lubricant, heating of the tool die is an attractive method. Chromium prealloyed powders are used in an increasing number of PM applications. These prealloyed Cr-powders are attractive as they combine high mechanical strength with cost effectiveness. However, due to the solution hardening effect of the prealloying elements, mixes based on prealloyed powders obtain lower compressibility compared to mixes based on pure iron powders. Therefore, Cr alloyed powder can be more demanding to compact and require more efficient lubricants. 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 is presented including compaction of multi level components under production conditions.

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 N₂/H₂ based sintering atmosphere with low partial pressure of oxygen, sintering of chromium alloyed sinter steels is feasible. By having chromium prealloyed 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 and mechanical properties are further enhanced if high temperature sintering is utilized. However, due to the solution hardening effect of the prealloying elements, mixes based on prealloyed 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.

In this paper two grades of prealloyed chromium steels were investigated, Astaloy CrL prealloyed with 1.5% Cr and 0.2% Mo and Astaloy CrM prealloyed with 3.0% Cr and 0.5% Mo. Two types of 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 three evaluations of the two types of mixes, covering different aspects of
the compaction 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. This evaluation also included a benchmark of the weight control of the parts
besides the compressibility and ejection force. 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.

First investigation: Compressibility and ejection performance
Four mixes were made for the first two investigations, see Table 1 for mix type, compositions
and designations. The Intralube® E mixes had 0.2% and 0.15% lower lubricant amount
respectively, compared to the corresponding amidewax Premix. The batch size of each mix
was 1 ton.

Table 1: Mixes

<table>
<thead>
<tr>
<th>Designation</th>
<th>Type of mix</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrL-P</td>
<td>Premix</td>
<td>Astaloy CrL + 1% Cu + 0.65% Gr. + 0.3% MnS + 0.8% Amidewax</td>
</tr>
<tr>
<td>CrL-I</td>
<td>Intralube® E mix</td>
<td>Astaloy CrL + 1% Cu + 0.65% Gr. + 0.3% MnS + 0.6% Lube E</td>
</tr>
<tr>
<td>CrM-P</td>
<td>Premix</td>
<td>Astaloy CrM + 0.55% Graphite + 0.8% Amidewax</td>
</tr>
<tr>
<td>CrM-I</td>
<td>Intralube® E mix</td>
<td>Astaloy CrM + 0.55% Graphite + 0.65% Lube E</td>
</tr>
</tbody>
</table>

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, 650 MPa and 800 MPa. The dimensions of the
rings were Ø55/45 mm and height 15 mm. 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 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 amidewax 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.

Tensile test bars according to ISO 2740 were compacted under similar conditions as the ring
shaped specimens. The test bars were sintered at 1120°C, 30 min in 90 N2/10 H2
atmosphere. The sintered density was measured by weighing the specimens in air and water.

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, a slice is cut out to show the cross section.
The outer diameter of the parts was 77 mm and the height of the outer segment was 21 mm.
The weight of the parts was ~470g, exact weight depending on the density level. The press
rate was 6.3 parts/minute.
For each mix, two compaction runs were made at compacting pressures of 440 MPa and 560 MPa respectively. The maximum compacting pressure was limited to 560 MPa since the capacity of the press, 2500 kN, was reached. As in the first investigation the tool die was not heated when the amidewax Premix parts were compacted while the tool die was heated to 70°C when the 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 (ISO standard 2738).

**Third investigation: Max height of bushings**

In the third investigation the same two types of mixes, amidewax 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>
<thead>
<tr>
<th>Designation</th>
<th>Type of mix</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrM-P2</td>
<td>Premix</td>
<td>Astaloy CrM + 0.5% Graphite + 0.6% Amidewax</td>
</tr>
<tr>
<td>CrM-I2</td>
<td>Intralube® E mix</td>
<td>Astaloy CrM + 0.5% Graphite + 0.6% Lube E</td>
</tr>
</tbody>
</table>

The dimensions of the bushings were Ø40/20 mm and height was varied in the range 25 – 50 mm. A 100 ton hydraulic Gasbarre press was used for the trials. The tooling had a carbide insert and the press tool had a floating core rod. The tool die was heated to 60°C in the trials with both lubricants. The evaluations were started with a height of 25 mm and 30 parts were pressed, thereafter the height was increased in increments of 2.5 mm 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 and 800 MPa.

**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 the amidewax Premix and Intralube® E mixes. The apparent density was slightly higher for the amidewax Premix while Flow was faster for Intralube® E mix in the case of the Astaloy CrL based mixes.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Type of mix</th>
<th>AD [g/cm³]</th>
<th>Flow [s/50g]</th>
<th>Graphite [%]</th>
<th>Lubricant [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrL-P</td>
<td>Premix</td>
<td>2.96</td>
<td>33.8</td>
<td>0.65</td>
<td>0.80</td>
</tr>
<tr>
<td>CrL-I</td>
<td>Intralube® E mix</td>
<td>2.91</td>
<td>31.8</td>
<td>0.68</td>
<td>0.62</td>
</tr>
<tr>
<td>CrM-P</td>
<td>Premix</td>
<td>2.83</td>
<td>34.3</td>
<td>0.58</td>
<td>0.83</td>
</tr>
<tr>
<td>CrM-I</td>
<td>Intralube® E mix</td>
<td>2.80</td>
<td>34.2</td>
<td>0.59</td>
<td>0.66</td>
</tr>
</tbody>
</table>
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 Astaloy CrM compressibility was somewhat lower compared to Astaloy CrL. 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 650 MPa as with the amidewax Premix at 800 MPa. 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. 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, see Figure 3.

In Figure 4 ejection energy is plotted versus the green density. CrL-I and CrM-I were significantly lower in ejection energy compared to the corresponding amidewax 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 energy of the Astaloy CrM mixes were higher compared to the Astaloy CrL mixes.

There was less difference between the two types of mixes in terms of static ejection force, see Figure 5. In the case of CrL-I the ejection force was lower compared to CrL-P when comparing at the same density level. For Astaloy CrM mixes ejection force was similar at 7.06 g/cm³ but at lower density level the ejection force was lower with CrM-P.
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>
<thead>
<tr>
<th>Comp. Pressure Level</th>
<th>Ejection Force Mean [kN]</th>
<th>Ejection Weight Mean [g]</th>
<th>Ejection Weight Std.dev [g]</th>
<th>Ejection Weight Std.dev [%]</th>
<th>Part Temp. [°C]</th>
<th>Green density [g/cm³]</th>
<th>Sintered density [g/cm³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrL-P Low</td>
<td>437</td>
<td>180</td>
<td>465.4</td>
<td>1.15</td>
<td>0.25%</td>
<td>52</td>
<td>6.72</td>
</tr>
<tr>
<td>High</td>
<td>550</td>
<td>217</td>
<td>484.8</td>
<td>1.13</td>
<td>0.23%</td>
<td>59</td>
<td>6.92</td>
</tr>
<tr>
<td>CrL-I Low</td>
<td>434</td>
<td>175</td>
<td>463.9</td>
<td>1.14</td>
<td>0.25%</td>
<td>62</td>
<td>6.72</td>
</tr>
<tr>
<td>High</td>
<td>548</td>
<td>183</td>
<td>483.1</td>
<td>1.06</td>
<td>0.22%</td>
<td>67</td>
<td>6.95</td>
</tr>
<tr>
<td>CrM-P Low</td>
<td>433</td>
<td>180</td>
<td>452.5</td>
<td>0.77</td>
<td>0.17%</td>
<td>56</td>
<td>6.60</td>
</tr>
<tr>
<td>High</td>
<td>562</td>
<td>238</td>
<td>477.9</td>
<td>0.84</td>
<td>0.18%</td>
<td>62</td>
<td>6.89</td>
</tr>
<tr>
<td>CrM-I Low</td>
<td>444</td>
<td>175</td>
<td>457.5</td>
<td>0.89</td>
<td>0.19%</td>
<td>63</td>
<td>6.66</td>
</tr>
<tr>
<td>High</td>
<td>557</td>
<td>192</td>
<td>482.9</td>
<td>0.92</td>
<td>0.19%</td>
<td>68</td>
<td>6.93</td>
</tr>
</tbody>
</table>

In Figure 6 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 CrL-I and CrM-I were significantly lower compared to the corresponding amidewax Premix. The temperature of the parts after ejection is presented in Figure 7. With higher compacting pressure the temperature of the parts after ejection was higher. The temperature of the parts made of CrL-I and CrM-I was, as can be expected, higher due to the heating of the tool die. The temperature difference between parts made of the Intralube E mixes pressed with heated tool die and the corresponding amidewax Premix was in the range 6°C to 10°C.

The weight scatter of the pressed parts is presented in Figure 8. The only significant difference was that the Astaloy CrM mixes scattered less than the Astaloy CrL mixes. Between the two types of mixes there was no significant difference in weight scatter of the pressed parts.
In Figure 9 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 influence 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. In Figure 10 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 before and after the sintering.

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 amidewax 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 the amidewax Premix 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 CrM-I2 and CrM-P2 had the same lubricant content, 0.6%, and the tool die was heated to 60°C during the compaction of both mixes.

In Figure 11 the height of the highest bushings with scratch free surface which were pressed of the two mixes at 600 MPa and 800 MPa are presented. At 600 MPa 20 mm higher bushings could be compacted with CrM-I2 compared to CrM-P2. At 800 MPa the difference in achievable height with the two mixes was 12.5 mm.
Conclusions
Based on the results where Intralube® E mixes based on chromium alloyed base powder were compared to amidewax Premix of the same nominal compositions, it has been demonstrated that:

- Flow and apparent density are comparable between Intralube® E mix and the amidewax containing Premix.
- 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 amidewax Premix.

Acknowledgment
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References