LUBRICANTS FOR COMPACTION OF P/M COMPONENTS

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Abstract
In the production of P/M components admixed lubricant in the metal powder mix is the most common way to achieve the lubrication needed during compaction and ejection of the components. However, the type of lubricant not only influences the compaction, but also the properties of the powder mix, as well as the properties of the green component. In this paper a comparison is made between four types of lubricants; zinc stearate, amide wax, Kenolube® and Metallub®. The properties of GS-lube, a lubricant designed for high green strength, are also presented.

Introduction
During the P/M compaction process, admixed lubricant in a metal powder mix has two aims; to reduce internal friction (friction between particles) and reduce external friction (friction between compact and die wall). Compressibility increases at lower compacting pressures as an effect of reduced internal friction due to addition of lubricant. At higher compacting pressures, compressibility is decreased by an increased amount of lubricant, due to the fact that the lubricant itself occupies volume and that its density is much less than that of the metal. Reducing external friction means less tool wear as well as less heat build-up on the die wall.

Admixing a lubricant in the iron powder also influences the powder properties, as well as the properties of the green compact. Generally, apparent density is at the same level or increased by the addition of lubricant. Flow time is generally increased by addition of lubricant.

Green strength is decreased to various degrees by the lubricant addition. Green strength is important for being able to handle the green components before sintering. If sufficiently high green strength can be obtained, it opens up possibilities for machining operations on the components in green state. This is of interest for high-strength materials, due to dramatically reduced tool wear.

Clean lubricant burn off is also a concern in order to avoid staining of the sintered component’s surfaces. Lubricants containing zinc may cause deposits in the inlet zone of the sintering furnaces.

General description of lubricants
At first glance numerous lubricants for cold compaction are available. However, these can be divided into three groups, namely:

1. Metal soaps
2. Amides
3. Composite lubricants

The first two groups of lubricants consist of one major component each. The third group includes lubricants that consist of composites of different chemical substances.
Metal soaps is one of the most established groups of solid lubricants for metal powders. The most commonly used metal soap is zinc stearate, which consists chemically of a zinc ion with two anions of stearic acid attached. Zinc stearate has a laminar crystal structure.

However, metal soaps have some disadvantages. One of them, deposits, is well known - these occur on the surface of the compacted parts after sintering and zinc compounds are also deposited in the inlet zone of the sintering furnaces. Another disadvantage, of course, is the environmental problems caused by zinc emissions during the burn off.

The second group of lubricants is designated as amide waxes. This means, from a chemical point of view, that they consist of a diamide with two fatty acid chains. The most common type of fatty acid chain is stearic acid.

In addition to the two groups mentioned above, there is a group of lubricants which represents a range of combinations of different chemical substances. The purpose of taking this step on from well-known one-component systems is to meet the special demands of different component manufacturing systems. Kenolube, Metallub and the experimental GS-lube are all composite lubricants that are designed to give unique properties.

Experimental

Comparison of lubricants
Average particle sizes, D50, of the lubricants were measured by laser diffraction.

Powder properties obtained with the four different lubricants were measured on mixes with a composition of Distaloy AE + 0.5% C-UF4 + 0.8% Lubricant. Distaloy AE is a diffusion alloyed powder based on a pure atomized iron powder with 4% Ni, 1.5% Cu and 0.5% Mo diffusion bonded. Mixes were prepared in a 5 kg Lödige plough-share mixer.

Flow and apparent density (AD) were measured according to ISO 4490 and ISO 3923 respectively. Green density (GD) was measured according to ISO 3927 on cylindrical specimens with diameter 25 mm and a weight of 50 g. Sintered density (SD) and ejection energy were measured on the same type of specimens. The ejection energy was calculated based on force and displacement data during pressing. Sintered specimens were cut and polished, and micrographs of the porosity were taken. Green strength was measured according to ISO 3995.

Green Machining
The purpose of the investigation was to evaluate green machining of parts compacted from a powder mix containing the experimental lubricant GS-lube compared to Amide Wax and a mixture compacted by die wall lubrication (DWL), i.e. without admixed lubricant to the iron powder. This was achieved through a quality test of drilled holes and photos of the outlet.

For the drill test, Ø80 mm discs pressed to a height of 12 mm and a density of 7.0 g/cm³ were used. Figure 1 shows the compacted disc with 16-drilled holes. A Ø5 mm drill was used for the tests and the feed rate was changed after every four holes drilled.
Edge Integrity
On the first compacted discs, a quality test was carried out on the drilled holes. The hole’s quality is defined by the edge integrity. The high-speed steel drill PFX A927 from Dormer was used for the quality testing. The cutting speed was 80 m/min, see table 1.

Table 1: Cutting speeds and feeds used in the quality tests of the compacted discs.

<table>
<thead>
<tr>
<th>Cutting speed (m/min)</th>
<th>Feed, ( f ) (mm/rev)</th>
<th>Rev (rpm)</th>
<th>Feed rate (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>0.025</td>
<td>5093</td>
<td>127</td>
</tr>
<tr>
<td>80</td>
<td>0.050</td>
<td>5093</td>
<td>255</td>
</tr>
<tr>
<td>80</td>
<td>0.100</td>
<td>5093</td>
<td>509</td>
</tr>
<tr>
<td>80</td>
<td>0.200</td>
<td>5093</td>
<td>1019</td>
</tr>
</tbody>
</table>

In a through-hole drilling operation there are two particular critical moments. The first one is when the drill tip enters the material, creating an inlet edge. The second one is when the drill tip leaves the material, creating an outlet edge on the other side of the disc. In the drilling operation, both the entering and leaving material causes variously damaged edges. To estimate the defect dimensions at the edges, the holes are scanned with a contour measurement machine (Mahr Contouroscop C4P). Figure 2 shows, as an example, a contour found during a scan.

Figure 2: Left picture: Schematic principle of scanning edge integrity with probe (needle). Right picture: Breakout of an inlet edge.
As this example shows, the appearance of the damage exhibits an irregular contour that in most cases consists of a combination of breakouts and burrs. In this paper edge e, depth of breakout at the edges under an angle of 45°, is presented. This is not a complete characterisation of the defects, which would also include hole x and face y, but still gives a good comparison of the defect size.

Photos of the outlet of the drilled holes are shown in this paper. The photos show the outlets of drilled holes with the lowest feed rate, 0.025 mm/rev, and the highest, 0.2 mm/rev. The compacted discs were drilled with a cutting speed of 70 m/min using a solid carbide R120 drill from Dormer. Table 2 shows the 4 different feed rates used in the drilling test.

Table 2: Cutting speeds and feeds for the second compacted and drilled discs.

<table>
<thead>
<tr>
<th>Cutting speed (m/min)</th>
<th>Feed, f (mm/rev)</th>
<th>Rev (rpm)</th>
<th>Feeding/(mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70</td>
<td>0.025</td>
<td>4456</td>
<td>111</td>
</tr>
<tr>
<td>70</td>
<td>0.050</td>
<td>4456</td>
<td>223</td>
</tr>
<tr>
<td>70</td>
<td>0.100</td>
<td>4456</td>
<td>446</td>
</tr>
<tr>
<td>70</td>
<td>0.200</td>
<td>4456</td>
<td>891</td>
</tr>
</tbody>
</table>

Results and discussion

Comparison of lubricants

In table 3 average particle size and Zn-content of the four lubricants are presented. As can be seen, there are big differences in particle size between the lubricants. Of the four lubricants, zinc stearate is the finest, while Metallub is the coarsest. A coarse particle size is beneficial for lubrication, but results in larger pores in the sintered component.

Table 3: Particle size and content of Zn

<table>
<thead>
<tr>
<th></th>
<th>Zinc Stearate</th>
<th>Amide Wax</th>
<th>Metallub</th>
<th>Kenolube</th>
</tr>
</thead>
<tbody>
<tr>
<td>D50</td>
<td>4 µm</td>
<td>7 µm</td>
<td>45 µm</td>
<td>20 µm</td>
</tr>
<tr>
<td>Zn</td>
<td>11%</td>
<td>-</td>
<td>10%</td>
<td>2%</td>
</tr>
</tbody>
</table>

Flow and apparent density of pure, unblended Distaloy AE are 24 s/50 g respectively 3.05 g/cm³. As can be seen in figure 3, all four lubricants increase flow time to various degrees. Amide wax is the lubricant that gives the longest flow time. Best flow is obtained for mixes with Metallub and Kenolube. When mixing the iron powder with lubricant, an increase in apparent density is obtained when zinc stearate, Kenolube or Metallub is used as lubricant, see figure 4. With Amide wax the apparent density of the mix is similar to the apparent density of the base iron powder. However, apparent density of mixes is also to some extent dependent on intensity of the mixer and mixing time.
Mixes with zinc stearate or Kenolube as lubricant obtain best compressibility, with higher green and sintered density as a result, see figure 5. Green strength is superior with Kenolube as lubricant, see figure 6. Amide wax gives slightly better green strength compared to zinc stearate and Metallub.

Ejection energy versus green density at three levels of compaction pressure (400, 600 and 800 MPa) is presented in figure 7. Low ejection energy is desired to minimize tool wear. At low compacting pressures the difference between the four lubricants is small, but at high compacting pressures the differences are significant. The mix with zinc stearate obtains poorest lubrication, while mixes with Kenolube and Metallub give best lubrication. Coarse particle size and composite structure are factors that contribute to the good lubricity of these lubricants.

With the very good lubrication and good compressibility obtained with Kenolube, lower amounts of lubricant are possible. At high compacting pressures, mixes with 0.6% Kenolube exhibit lower ejection energy compared to a mix with similar composition, but with 0.8% zinc.
stearate as lubricant. With this decreased amount of lubricant, density is further increased at high compacting pressures.

![Graph showing ejection energy vs. green density of mix with Distaloy AE + 0.5% C + 0.8% Lubricant](image)

**Figure 7: Ejection energy vs. green density of mix with Distaloy AE + 0.5% C + 0.8% Lubricant**

A drawback with coarser particle size of lubricants is larger pores in the sintered structure of the component. Micrographs of the pore structure of sintered materials based on the mixes with the four lubricants are shown in figure 8. With Metallub, large pores, due to the coarse particle size of the lubricant, are clearly seen. In applications where fatigue strength is of importance, size of the largest pores may be a limiting factor.

![Micrographs showing pore structure](image)

- Amide Wax, X50 = 7µm
- Zinc stearate, X50 = 4 µm
- Kenolube, X50 = 20 µm
- Metallub, X50 = 45 µm

**Figure 8: Difference in porosity of mix with Distaloy AE + 0.5% C + 0.8% Lubricant compacted at 600 MPa**
Lubricant for increased Green Strength - Powder and Green Properties

The results from the Hall Flow and AD, comparing mixes with the experimental lubricant GS-lube and amide wax respectively, are shown in table 4. The mixture containing GS-lube has a better flow than amide wax, but the apparent density is slightly lower.

Table 4: Powder properties of mixes with a composition of Distaloy AE + 0.6%C + 0.8% Lubricant.

<table>
<thead>
<tr>
<th>Powder Properties</th>
<th>Experimental lubricant GS-lube</th>
<th>Amide Wax</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow (s/50g)</td>
<td>28.7</td>
<td>30.9</td>
</tr>
<tr>
<td>Apparent Density (g/cm³)</td>
<td>2.97</td>
<td>3.02</td>
</tr>
</tbody>
</table>

Green and sintered properties of the same two mixes are presented in table 5. The main benefit of a mix with GS-lube is the very high green strength of pressed components, while the disadvantages are poorer lubrication, indicated by the higher ejection energy, and the lower green and sintered density compared to the mix with amide wax.

Table 5: Green and sintered properties at a compaction force of 600 MPa of mixes with a composition of Distaloy AE + 0.6%C + 0.8% lubricant

<table>
<thead>
<tr>
<th>Property</th>
<th>Experimental lubricant GS-lube</th>
<th>Amide Wax</th>
</tr>
</thead>
<tbody>
<tr>
<td>Green Density (g/cm³)</td>
<td>7.01</td>
<td>7.06</td>
</tr>
<tr>
<td>Green Strength (MPa)</td>
<td>26.1</td>
<td>12.6</td>
</tr>
<tr>
<td>Rattler (%)</td>
<td>0.23</td>
<td>0.69</td>
</tr>
<tr>
<td>Ejection energy (J/cm²)</td>
<td>38.6</td>
<td>32.9</td>
</tr>
<tr>
<td>Sintered Density (g/cm³)</td>
<td>6.97</td>
<td>7.04</td>
</tr>
</tbody>
</table>

Table 6 shows the green density and green strength of the mix without any lubricant when compacted to nearly the same density as the mixes with GS-lube and amide wax. By excluding the lubricant, the green strength will increase by 76% compared to a mix with amide wax.

Table 6: Green density and green strength of a mix with composition Distaloy AE + 0.5% C compacted with die wall lubrication (DWL) to a density of 7.0 g/cm³

<table>
<thead>
<tr>
<th>Powder Properties</th>
<th>DWL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Green density (g/cm³)</td>
<td>6.97</td>
</tr>
<tr>
<td>Green Strength (MPa)</td>
<td>22</td>
</tr>
</tbody>
</table>

The green strength of a mix with experimental GS-lube is superior compared to amide wax. The low rattler value is also a considerable benefit of using this lubricant. A low rattler value is very important to prevent components from getting damaged during handling and transport between press and sintering furnace. Figure 9 shows that the green strength was increased by over 100% and that the weight loss is 67% less than with amide wax.
Figure 9: Green strength and loss of weight (Rattler) at compacting pressure 600 MPa with the experimental lubricant GS-lube and Amide wax.

This concept is very suitable for components with a complex geometry or components that are green machined before sintering. The concept is also suitable for components with a low density \(\sim 6.0 \text{ g/cm}^3\), the production of bearings for example. At such low green densities, high green strength is an important requirement.

Edge Integrity
The results of the measurements of edge integrity are plotted as curves in feed rate vs. mean breakout, see figures 10-11. The depth of breakout at the edges under an angle of 45\(^\circ\) is shown for both the inlet and outlet. With both materials, the sizes of breakouts at the entering and outlet edges increase with an increasing feed level.

The dimension “e” is much lower for a mix with GS-lube compared to amide wax, which is a considerable advantage. These results show that there are greater possibilities for green machining of components compacted from a mix with GS-lube than amide wax. Irrespective of the lubricant used, the best edge integrity is obtained at a feed rate between 0.025 mm/rev and 0.050 mm/rev.

Photos were taken of the outlet edge of the drilled holes. Figure 12-17 shows the outlets of drilled holes with the lowest feed, 0.025 mm/rev, and the highest, 0.2 mm/rev. Figures 14 and 17 show the outlet edge on a disc compacted with DWL from a mix without any lubricant. The composition of the mix is Distaloy AE with 0.6% graphite.
The photos show the benefit of the experimental GS-lube both at the lowest speed, 0.025 mm/rev, and the highest, 0.2 mm/rev. The photos also show that the breakout becomes worse when the feed level increases. It is also seen that it is better to use amide wax as a lubricant rather than no lubricant at all, even though the green strength is lower for a mix containing lubricants.

Figure 12: Outlet edge of a mixture with experimental GS-lube. Feed level = 0.025 mm/rev

Figure 13: Outlet edge of a mixture with Amide Wax. Feed level = 0.025 mm/rev

Figure 14: Outlet edge of a mixture with no lubricant. Feed level = 0.025 mm/rev

Figure 15: Outlet edge of a mixture with experimental GS-lube. Feed level = 0.2 mm/rev

Figure 16: Outlet edge of a mixture with Amide Wax. Feed level = 0.2 mm/rev

Figure 17: Outlet edge of a mixture with no lubricant. Feed level = 0.2 mm/rev
Discussion
The principal benefits of zinc stearate are good powder properties with fast flow and high apparent density. High green and sintered density is obtained together with small pores. Its disadvantages are high ejection energy and the zinc content of the lubricant, which may cause stained components, and Zn accumulation in the furnace. Mixes with zinc stearate as lubricant also form more dust than mixes with the other lubricants.

Amide wax has the advantage of smaller pores as compared to the other lubricants due to the small particle size. Since amide wax is purely organic, it has very good burn off characteristics. The apparent density is rather low and powder flow is slower than the other lubricants.

Kenolube combines the good powder properties of zinc stearate with lubrication that surpasses amide wax. Green strength obtained with Kenolube is significantly higher compared with both zinc stearate and amide wax.

Metallub has the advantages of very good lubrication, high apparent density and good flow. On the other hand it results in rather large pores, low green and sintered density.

The green strength of a mixture with the experimental lubricant GS lube is very high. By using GS-lube instead of amide wax, the green strength will increase by over 100%. Better edge strength is demonstrated by the low Rattler value. Using a mix with GS-lube substantially increases possibilities to carry out green machining and production of complex parts with no cracks.

The lubricity of the experimental lubricant GS-lube is lower than traditional lubricants resulting in somewhat lower density and higher ejection force. GS-lube is also suitable for components with a low density ~6.0 g/cm³, the production of bearings for example.

Excluding the lubricant in the powder mix and utilize die wall lubrication increase both the green density and green strength. Despite this, it is difficult to carry out green machining because extensive damage occurs on components. These results show that a lubricant must be used in a powder mix for green machining.

References