

# Lubricant distribution on compacts and tool walls after P/M compaction

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The use and effect of lubricants during compaction and ejection of metal powder has for a long time been studied from a macro perspective, assessing effects of different factors but ignoring the actual mechanisms through which they operate. Here, the intention is to explain the physical interaction between the metal powder, lubricant and tool wall on a microscopic level. With the development of a new compaction tool, the use of X-ray photoelectron spectroscopy and ion sputtering, it has been established that, for iron powder samples containing 0.5-3 wt% ethylene bis-stearamide, compressed with 400-800 MPa, both the compact surface and the tool wall are to a large extent covered by a film of monomolecular thickness. Through interference microscopy, the volume of pores at the surface of the compact have been determined to  $0.4 (\mu\text{m})^3/(\mu\text{m})^2$ . Several conclusions that deepen the understanding of the functionality of lubricants have been drawn from these observations.

## Introduction

The primary role of lubricants in the powder metallurgy (P/M) industry is to facilitate the compaction process by reducing the friction between the powder and the die wall, both during compaction of the powder and during the ejection of the green part. Today these lubricants are not only beneficial but are also the cause for several problems as they limit green density, may cause contamination of furnace and green part, can reduce green strength, etc. There is therefore a continuous need to develop new and better lubricants in order to fight these problems more efficiently. Development has so far mainly been performed by looking at powder compaction and the lubricants effect from a “macro” perspective. The choice of lubricant and the amounts used are usually empirically optimised by evaluating parameters such as density, strength, ejection pressure, etc [1-5]. As development continues it has become more and more relevant to adopt a “microscopic” perspective instead, and attempt to explain the physical and chemical interaction between the powder, lubricant and the tool wall

during the compaction step in detail. Fundamental understanding at this level is expected to open up new possibilities to develop more efficient lubricants in the future.

This interaction, during compaction, is expected to involve both chemical and physical processes. The physical interaction consists of all effects that result from the physical displacement of the components in the powder mixture (i.e. lubricant and metal powder particles). Examples of this type are migration of lubricant within the powder mix/green body, formation of a lubricant film between the tool wall and the green compact during compaction and displacement of lubricant in the near surface region during ejection. Chemical interaction, on the other hand, includes reactions between lubricant and metal particles (and the surrounding atmosphere) during compaction and ejection, as well as the degradation of lubricant because of high temperatures or shear stresses. The friction reducing effect that is the primary reason for adding a lubricant is expected to mainly be due to the physical interaction. Friction is reduced as the more easily deformable lubricant physically separates the compacted powder part and the compaction tool wall. In this work we have chosen to disregard chemical effects and instead focus on investigating the physical interaction. This was done for practical reasons. Since the physical effects are an essential part of the lubrication process and neither of the two mechanisms has previously been described, separating them was seen as necessary in order to avoid interaction effects.

## Experimental Procedure

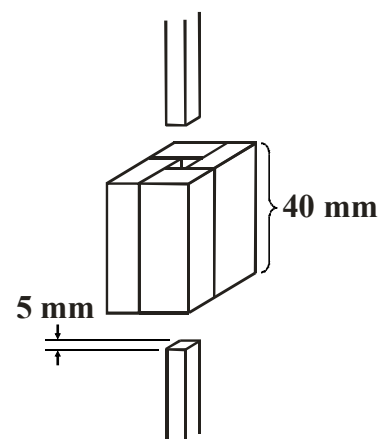
### Material

Powder mixtures were prepared with water atomised iron powder, HÖGANÄS ASC 100.29 from Höganäs AB and ethylene bis-stearamide (EBS) lubricant. The lubricant had a bulk density of  $1.0 \text{ g/cm}^3$  and mean particle size of  $5,5 \text{ }\mu\text{m}$  measured by laser diffraction technique. During studies of green powder compacts, lubricant content in the range of 0.5 to 3 wt% was used. The studies of the lubricant distribution on the tool wall were performed with 0.8 wt% EBS in the powder mixture. No graphite was added to the powder.

### Compaction Tools

The first compaction tool was of conventional type and the produced compacts had geometry of  $30 \times 12 \times 6 \text{ mm}^3$  (normally used for green strength measurements, ISO 3995:1985). Analysis of the compact surface was made on these compacts. The second compaction tool (figure 1) was used for analysis of the tool wall. It consisted of 4 rectangular tool sections of hard metal with the dimensions  $40 \times 13 \times 8 \text{ mm}$ , forming a  $5 \times 5 \text{ mm}$  cavity. These sections were locked into position by screws through the surrounding matrix.

**Figure 1.** The compaction tool setup with upper and lower punches and 4 tool sections forming the cavity. The matrix surrounding the 4 sections is not shown.



### Interference microscopy

An optical profilometer (RST WYKO) was used in this investigation. It can construct 3 dimensional images of surfaces up to a depth of  $500 \text{ }\mu\text{m}$  with a resolution better than  $10 \text{ nm}$ . With a magnification of 40:1 the area analysed in each measurement was  $150 \text{ }\mu\text{m} \times 100 \text{ }\mu\text{m}$  with a standard pixel size of  $0.4 \text{ }\mu\text{m} \times 0.45 \text{ }\mu\text{m}$ .

## SEM

SEM images were taken with a Zeiss DSM 940A instrument at magnifications of 300:1 and 1000:1 using beam energy of 10 kV. Samples were gold plated with an approximately 50 Å thick layer prior to analysis with a Sputter Coater S150B. Image analysis on SEM images was performed with Optimas 6.1 Software.

## Debinding procedure

Samples selected for debinding were kept at 450 °C for 40 minutes in 100 % hydrogen atmosphere. They were then sintered for 10 minutes at 700 °C in 90 % nitrogen, 10 % hydrogen atmosphere to improve mechanical properties. After cooling, the samples were washed repeatedly (60, 30 and 15 min) with trichloroethylene and ultrasonic vibrations in order to remove all residues of organic material.

## XPS and Ion sputtering

A PHI 5500 instrument with monochromatic Al K $\alpha$  radiation ( $h\nu=1486.6$  eV) was used in this investigation. Spectra were acquired at 23.5 eV pass energy. Evaluation of XPS results was done with PHI Multipak and QUASES softwares. The aperture in front of the electron analyser was spherical with a diameter of 0.8 mm. Ion sputtering was performed with an argon source and beam energy of 4 kV, a total current of 9.2  $\mu$ A and a current density of 97 nA/mm<sup>2</sup> within the ion beam. On thick homogenous samples, the sputtering rate was determined to 35 Å/min for a 0.1  $\mu$ m layer of Ta<sub>2</sub>O<sub>5</sub> and to 30 Å/min for a layer of a metal-organic compound (C<sub>27</sub>H<sub>18</sub>AlN<sub>3</sub>O<sub>3</sub>) with the same thickness. The sputtered area was 5x5 mm<sup>2</sup>.

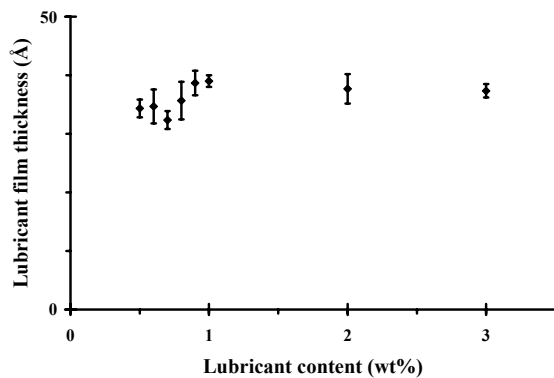
## **Results**

### XPS analysis of green compacts

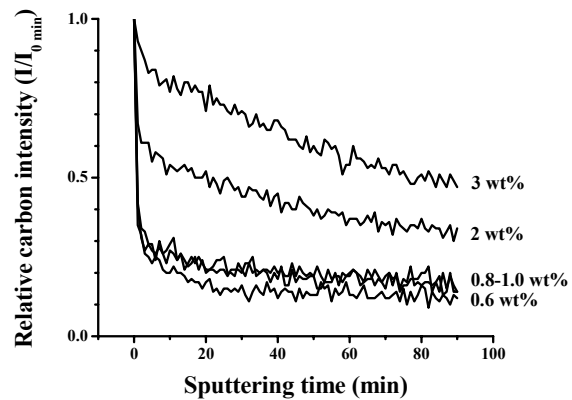
The thickness of the lubricant layer has been analysed as a function of lubricant content (0.5 to 3 wt%), compaction pressure (400-800 MPa) and number of previous compactions after the mounting of the compaction tool (1 to 20). Under all these conditions, the lubricant film remains stable with a thickness of below 50 Å. In figure 2 the thickness is shown for lubricant contents between 0.5 and 3 wt% in the powder mixture. This result indicates that the increased volume fraction of lubricant with increased amounts of lubricant in the mixture is not located at the contact area between the green part and the tool wall, but in pores and irregularities at the compact surface thicker than 100 Å, the maximum penetration depth of the method.

In order to investigate these thicker areas further green compacts were subjected to ion sputtering and sequential XPS analysis. The result from this investigation is shown in figure 3, expressed as the relative decrease of the carbon signal from the lubricant, as a function of sputtering time. Initially it decreases very rapidly (for lubricant contents between 0.6 and 1 wt% more than half of the total reduction in carbon intensity takes place during the first minute of sputtering) but the decrease rate is later reduced and the intensity reaches a constant value for lubricant contents between 0.6 and 1 wt%. At this stage the entire contact area is expected to be free from lubricant residues and the remaining carbon signal being emitted from lubricant filled pores, which are so deep (> 0.4  $\mu$ m) that the sputtering is not affecting the carbon intensity.

Due to the rapid initial decrease in carbon intensity we also assume that the entire area in contact with the tool wall, for lubricant contents of 1 wt% and below, is covered by a monolayer of EBS molecules. Even for higher lubricant contents a significant fraction of the contact area will have a similar thickness. The rapid initial decrease can be explained as the result of entire EBS molecules leaving the surface due to the ionic bombardment and their weak chemical bonding to the compact surface.



**Figure 2.** The thickness of the lubricant layer assessed by analysis [6, 7] of the XPS spektrum. For all lubricant contents, the thickness remains stable around 35 Å, a level slightly lower, but within the same range as the length of an outstretched EBS molecule (50 Å). Each value displays the average from three measurements.

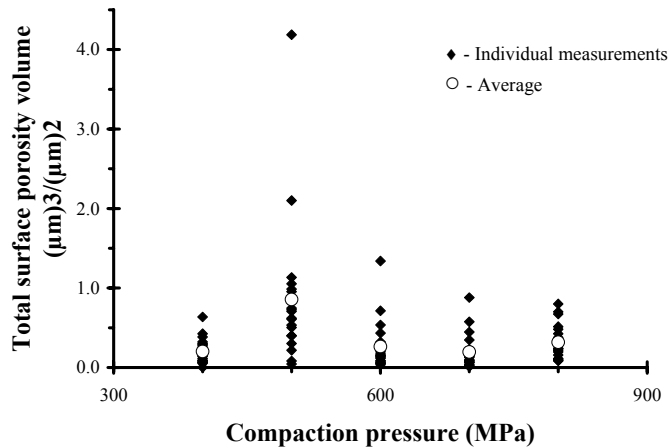


**Figure 3.** Relative carbon intensity from compacts with lubricant contents 0.5-3 wt%, subjected to ion sputtering. All levels of lubricant display an initial rapid decrease in carbon intensity during the first minute. This is an indication that entire molecules of EBS are being sputtered away from the compacts.

#### Interference microscopy analysis

Initial studies [6] have indicated that filling the pores located at the surface (here termed *surface porosity*) is a requirement in order to create the lubricant film at the contact area, which, in turn, is a requirement in order to achieve good lubrication. Interference microscopy measurements were carried out in order to estimate this volume. The result, displayed in figure 4 as the measured volume divided by the size of the analysed area, show average porosity volumes in the 0.2-0.9  $(\mu\text{m})^3/(\mu\text{m})^2$  range. The average porosity volume over all measured compaction pressures is 0.4  $(\mu\text{m})^3/(\mu\text{m})^2$ . The scattering in the results is caused by the natural porosity variation on a powder compact and the necessity to keep a small analysis area in each measurement (100  $\mu\text{m}$  x 150  $\mu\text{m}$ ) to ensure good accuracy.

The size of the detected porosity volume is, for all observed compaction pressures, remarkably small. The requirement to achieve lubrication is assumed to be creation of a monolayer film on the contact area (with a negligible volume) and filling the surface porosity. The small volume is an indication that the lubricant particles in direct contact with the tool wall before compaction, and within a short distance from it (within a few  $\mu\text{m}$ ), alone are sufficient in order to fulfil this requirement. For a homogenous powder mixture with 0.8 wt% lubricant and an apparent density of 3.0  $\text{g}/\text{cm}^3$ , the necessary amount of lubricant can be found within a 15  $\mu\text{m}$  wide layer of filled powder.

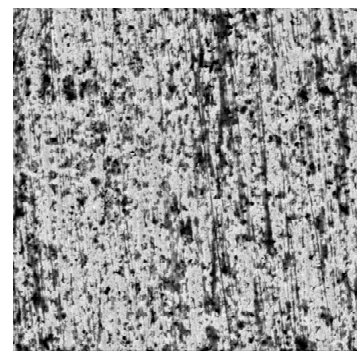


**Figure 4.** Volume surface porosity divided by the analysed surface area (100 µm x150 µm), as a function of compaction pressure. Each measurement point is an average of 20 randomly placed observations on the compact surface.

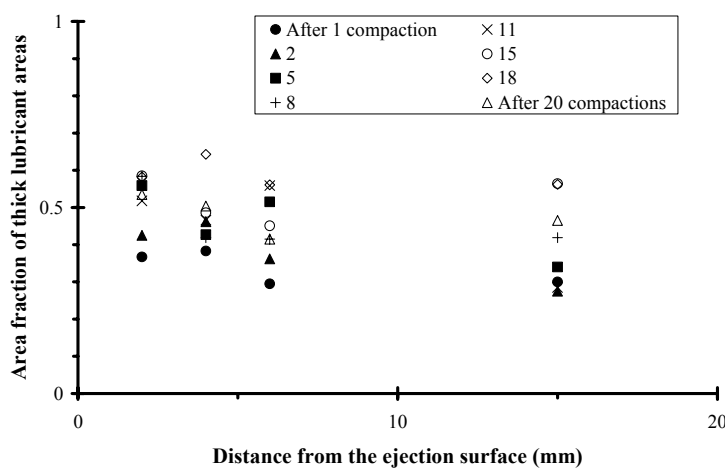
SEM and XPS analysis of the tool wall

With the construction of the new compaction tool, both SEM and XPS observations of the tool surface have been made possible. In an initial study, the tool surfaces were studied after 1-20 compactions at 400 MPa using powder mixtures with 0.8 wt% EBS. The SEM images have indicated an inhomogeneous lubricant distribution at the tool surface (figure 5). Thicker lubricant areas can be distinguished from the lighter hardmetal tool due to the difference in atomic number contrast between carbon and tungsten. Image analysis (figure 6) show that the covered fraction varies around 0.5 for all observed areas but it can be seen to increase slightly with increased number of compactions.

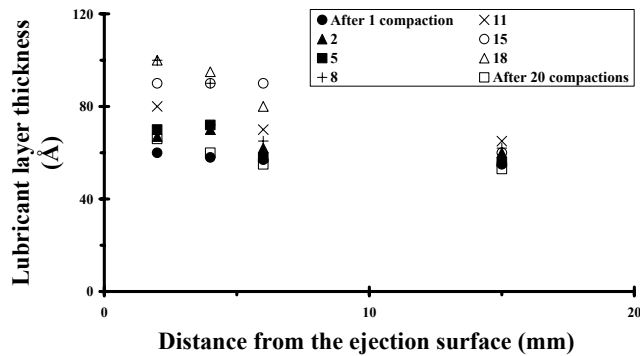
The lighter areas are also covered by a lubricant layer, thin enough to be analysed by XPS (figure 7). The thickness of this layer is thicker than comparable measurements on the powder compacts but it is still in the molecular monolayer range. The increased thickness could be an artefact caused by the 10 Å oxide layer covering the compaction tool beneath the lubricant. Investigation aimed at determining this is in progress. Clear differences can be seen between the constant thickness in the compaction area (at 15 mm) and the continuously increasing thicknesses closer to the ejection surface (2-6 mm).



**Figure 5.** SEM image over a 0.1x0.1 mm<sup>2</sup> area subjected to 5 compactions.



**Figure 6.** The area fraction of darker areas in SEM images corresponding to areas with thicker amounts of lubricant, for several positions on the tool surface. There is a general increase in coverage as the number of compacted parts increase.



**Figure 7.** Thickness of lubricant layer at 5 different positions on the tool wall after 1 to 20 compactions. The thickness increase within the compaction zone (10-20 mm) is negligible, in contrast to the increase in the ejection zone (0-10 mm).

## Conclusions

The main objective of this study has been to establish methods enabling microscopic observations of the lubricant distribution, both on compacted powder samples and on the tool surface. With the development of a new compaction tool, it has been possible to determine;

- Coverage of thick lubricant areas on the tool wall by SEM
- Thickness of thin lubricant films on the tool wall by XPS
- Thickness of lubricant areas on the powder compacts by XPS
- Surface porosity volume by optical interference microscopy

The continuing studies of these variables under different compacting conditions are expected to give deeper understanding of lubricant behavior during compaction. Already at this stage it can be of interest to note that:

- The volume of lubricant located on the surface is very small, around  $0.4 (\mu\text{m})^3/(\mu\text{m})^2$ . Only lubricant particles in direct contact with the tool wall before compaction or within a few  $\mu\text{m}$  from it seem to be causing the lubricating effect.
- Large fractions of both powder compacts and the tool surface are covered by a very thin lubricant film ( $< 100 \text{ \AA}$ ), which is in same range as the size of the lubricant molecule.

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