Presented at the Euro PM2015 congress held in Reims, France / October 2015 and published in the congress conference proceedings available from the European Powder Metallurgy Association (EPMA).

Manuscript refereed by Professor Paul Beiss (RWTH Aachen University, Germany)

Influence of the Lubricant Particle Size and Concentration on the Ejection Behaviour at Various Compaction Rates

Vincent Paris (Rio Tinto Metal Powders, 1655 Marie-Victorin Rd, Sorel-Tracy, QC, Canada, J3R 4R4) vincent.paris@riotinto.com;
Yannig Thomas (National Research Council Canada, 75 De Mortagne Blvd, Boucherville, QC, Canada, J4B 6Y4) yannig.thomas@cnrc-nrc.gc.ca;
Philippe François (Rio Tinto Iron & Titanium GmbH, Mergenthaleralle 77, 65760 Eschborn, Frankurt am Main, Germany) philippe.francois@riotinto.com;

Abstract

The development of new lubricants for single compaction requires an excellent understanding of the lubrication mechanisms, with the goal of optimizing their performance. The lubricant particle size distribution was already shown to have a significant impact on the ejection behaviour. This paper focuses on the more complex effects of the particle size distribution of the lubricant through the evaluation of the compaction and ejection performance given different concentrations of a mixture of fine and coarse lubricants and with various compaction parameters. FC-0208 powder mixes with different lubricant particle size distributions and concentrations were evaluated on an industrial mechanical press operating at various stroke rates and die temperatures. The ejection behaviour was recorded and compared to other parameters such as the springback and the green density.

Introduction

In the powder metallurgy process, lubrication is a key parameter. Several methods exist to provide lubrication, two of the most common being the admixing of an internal lubricant and the die wall lubrication technology with the former being by far more common. This approach is convenient due to the assurance that the compaction tools will always benefit from some level of lubrication and it is also simpler to use since it does not require additional equipment. However, internal lubrication has major drawbacks, especially if higher green densities are desired. The volume fraction occupied by the lubricant in a standard powder metallurgy mix is important. In an un-compacted volume of powder of standard formulation (FC-0208 MPIF designation) lubricated with 0.7% (by weight) of wax, 4.9% of the volume is occupied by the lubricant. During compaction, the lubricant will tend to limit the maximum achievable density. This explains why so much effort is focused on decreasing the amount of lubricant added to high performance powder mixes [1]. Die wall lubrication can address this issue by applying the lubricant only on the die surface, but its practical limitations (slowing of compaction cycle, suction-fill not being possible and the risk of a failed lubricant application) have so far limited its commercial use eventhough efficient die wall lubrication systems are now commercially available on the market [2].

A possible approach to reduce the amount of admixed lubricant and improve lubrication is the increase of lubricant migration through the powder volume as the compaction occurs and subsequent exit of the lubricant from the part at the die wall interface, where it is most needed for the soon-to-follow ejection stroke. There are several phenomena that occur in a stepwise fashion during the compaction cycle. Before compaction, the most abundant constituent in a powder volume is not the powder itself: nearly 60% of the volume is occupied by air, found in the voids between the powder particles. At the onset of compaction, particles will tend to rearrange themselves in the first place while some of the air is expelled from the compact. As the pressure increases, elastic and plastic deformation will eventually occur, a process which will end with the consolidation of the powder volume into a finished part. How the lubricant will react in those critical seconds between re-arrangement and full consolidation has been little studied [3, 4]. Once pressure is released, most compacted parts exhibit some level of elastic relaxation, commonly known as springback.

Lubrication mechanisms during compaction involve both chemical and physical processes. Physical processes are for instance 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 [4]. Chemical processes, on the other hand, include reactions between lubricant and metal particles during compaction and ejection, as well as the modification of lubricant properties because of high temperatures or shear stresses. The objectives of this paper are to investigate the effect of the particle size of lubricant as well as the compaction stroke rate which are expected to have an impact on the deformation and migration of lubricant towards the die wall. Studies in the field of compaction of pharmaceutical components have demonstrated that higher compaction speeds lead to increased ejection forces [5]. On the other hand, with metal powders, Mallender [6], using a pin-on-disk system, as well as Bonnefoy [7] using a shear plate device showed there was little or no effect on friction by increasing the sliding velocity. Other research demonstrated the beneficial impact of larger lubricant particle size on the ejection performance [8, 9].This paper attempts to describe both the influence of the physical and chemical properties of the lubricant and the compaction velocity on the ejection behaviour.

Experimental Procedure

A set of five steel powder mixes of 20 kg each were prepared using 0.7 wt% of various fatty acid based lubricants having different particle sizes and melting points (T_m). Ethylene bis-stearamide, a common powder metallurgy lubricant often referred to as EBS ($T_m \cong 145°C$) and a low melting point fatty acid based wax ($50°C < T_m < 100°C$). The detailed composition of the samples is provided in Table 1. For each powder sample, compaction and ejection behaviour was evaluated on a 150 mt Gasbarre mechanical press at the National Research Council Canada, Boucherville (Quebec). This press is equipped with a tungsten carbide (WC-Co) die, 25.4 mm in diameter with a core pin of 14.2 mm in diameter and strain gauges to monitor the compaction and ejection forces. Rings about 25.4 mm tall were compacted at different stroke rates of 5, 9 and 13 parts per minute which corresponds to punch speeds (in the compaction portion of the cycle) of 46, 94 and 142 mm/s, respectively. The M/Q ratio of the test part, defined as the lateral area over compacted area [1], was 9.08 as compared to 1.4 for a 6.35 mm (0.25 in) standard TRS bar, confirming the high friction area of the test part.

The green density of the parts was measured by weighing the parts and measuring their respective volume with a CMM apparatus, model Smartscope Flash 300 from OGP Inc., with a precision of 1.5 µm. The temperature of the parts was measured immediately after compaction using a type K, contact thermocouple.

Table 1. Detailed composition of the test samples.

Mix	Base Powder	Copper	Graphite	Lubricant (0.70 wt%)
1	AT-1001HP	1.80 wt% 165Q	0.70 wt% F-25	A
2				B
3				A+B
4				A+C
5				A+B+C

A: Fine EBS; B: Coarse EBS; C: Coarse low melting point wax ($50°C < T_m < 100°C$)

Results

Room Temperature Compaction

The graphs shown in Figure 1 present some of the results from room temperature compaction. The compaction stroke rate was 5 parts per minute (leftmost column), 9 parts per minute (centre column) and 13 parts per minute (rightmost column). For ease of comparison, all axes were set to the same scale. A coarse, high melting point EBS lubricant (B) tends to decrease the maximum achievable green density, regardless of the compaction pressure applied. However, using a portion of finer EBS or a low melting point lubricant can mitigate the compressibility limitation. A strong influence of the compaction rate was noted. At about 800 MPa, the maximum achievable green density gradually diminished as the compaction rate increased. Most lubricant lost about 0.03 g/cm^3 of density going from 5 to 13 parts per minute. The compaction rate also showed an effect on the springback. At 5 parts per minute, all lubricants contributed to a maximum radial springback between 0.22% and 0.27%. With the compaction speed increased to 9 parts per minute, the radial springback also increased, varying from 0.27% to 0.31% (at around 800MPa). The largest springback was observed at the highest compaction speeds and pressures with values exceeding 0.33%. While the nature of the lubricant contributed to some differences in the springback recorded for the compaction at a low stroke rate, very few differences are observed at the highest compaction speed.

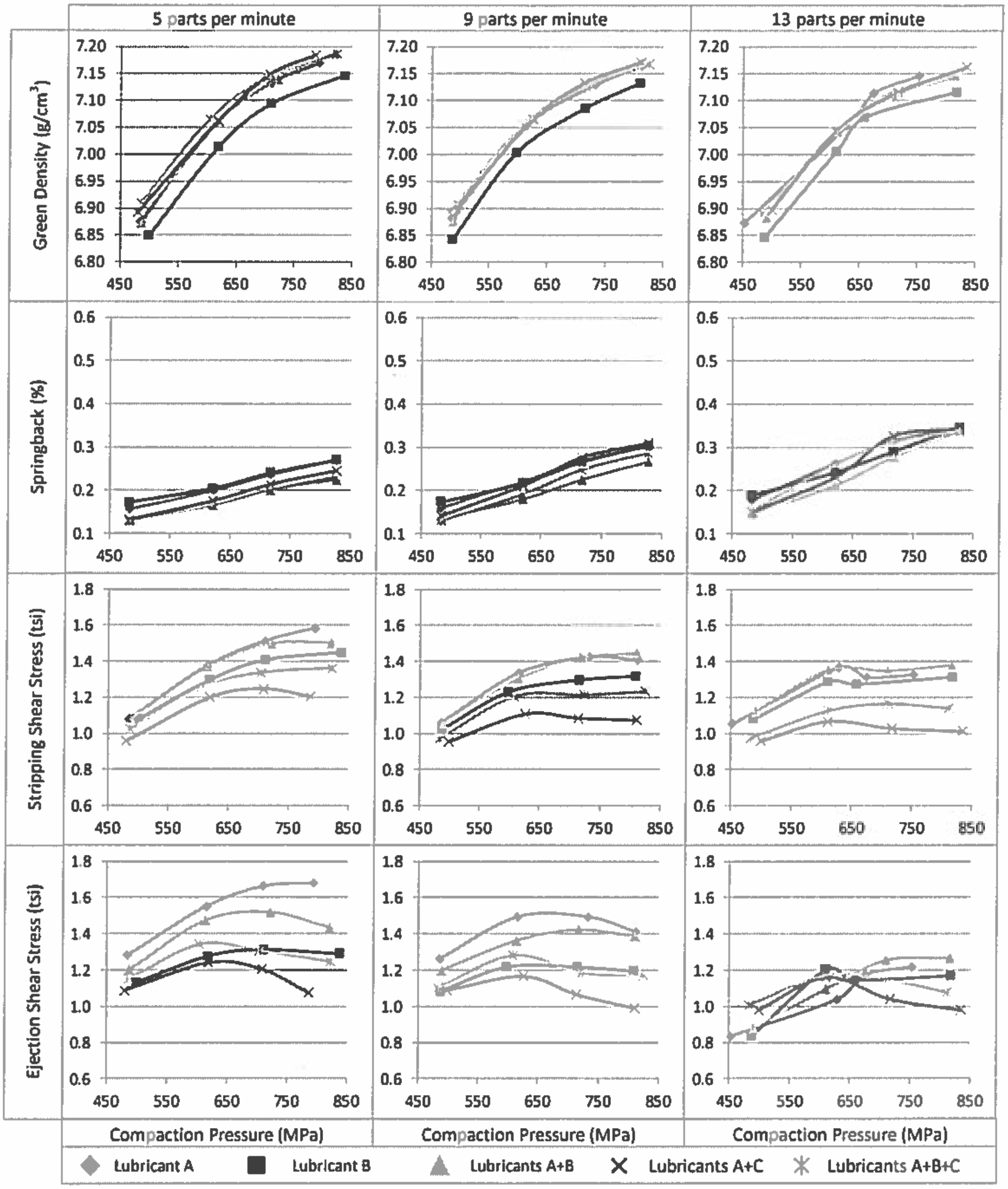

Figure 1. Results of the compaction performed with the non-heated die (ambient temperature).

Stripping and ejection shear stresses are a good approximation of the ejection behaviour. At the onset of the ejection process, forces peak sharply before the part begins to move out of the die and this force is computed into the stripping shear stress. As the part exits the die, the ejection force rapidly declines, due to the reduction in the surface area in contact with the die and this is calculated as the ejection shear stress. The nature and particle size distribution have a clear effect on the ejection behaviour. At the lower compaction speeds of 5 and 9 parts per minute and high compaction pressures, the impact of the increase in the particle size of the lubricant is obvious. As an example, with a 5 parts per minute stroke rate and at 827 MPa, the fine EBS recorded a stripping shear stress of 1.58 tsi, the mixture of fine and coarse EBS, 1.50 tsi and the coarse EBS, 1.45 tsi. The presence of low melting point lubricants further enhanced the ejection as demonstrated by the lower stripping and ejection shear stresses. As the compaction speed is increased, the benefits of both the larger particle size distribution and the low melting point lubricant are less obvious. This is demonstrated by the changes in the stripping shear stress values. At 5 parts per minute, the impact of both the coarser lubricant particle size and the low melting point lubricant are clearly distinguishable. The stripping shear stresses decreased with increasing particle size distribution and decreased even further with the addition of a low melting point lubricant. At 13 parts per minute, however, all three EBS mixes had comparable stripping shear stresses while the samples containing the low melting point lubricant still ejected better. The ejection shear stress values showed a similar trend but at 13 parts per minute, the results were much more variable, indicating that the effect of the particle size of the lubricant and its chemical nature had little influence on this value.

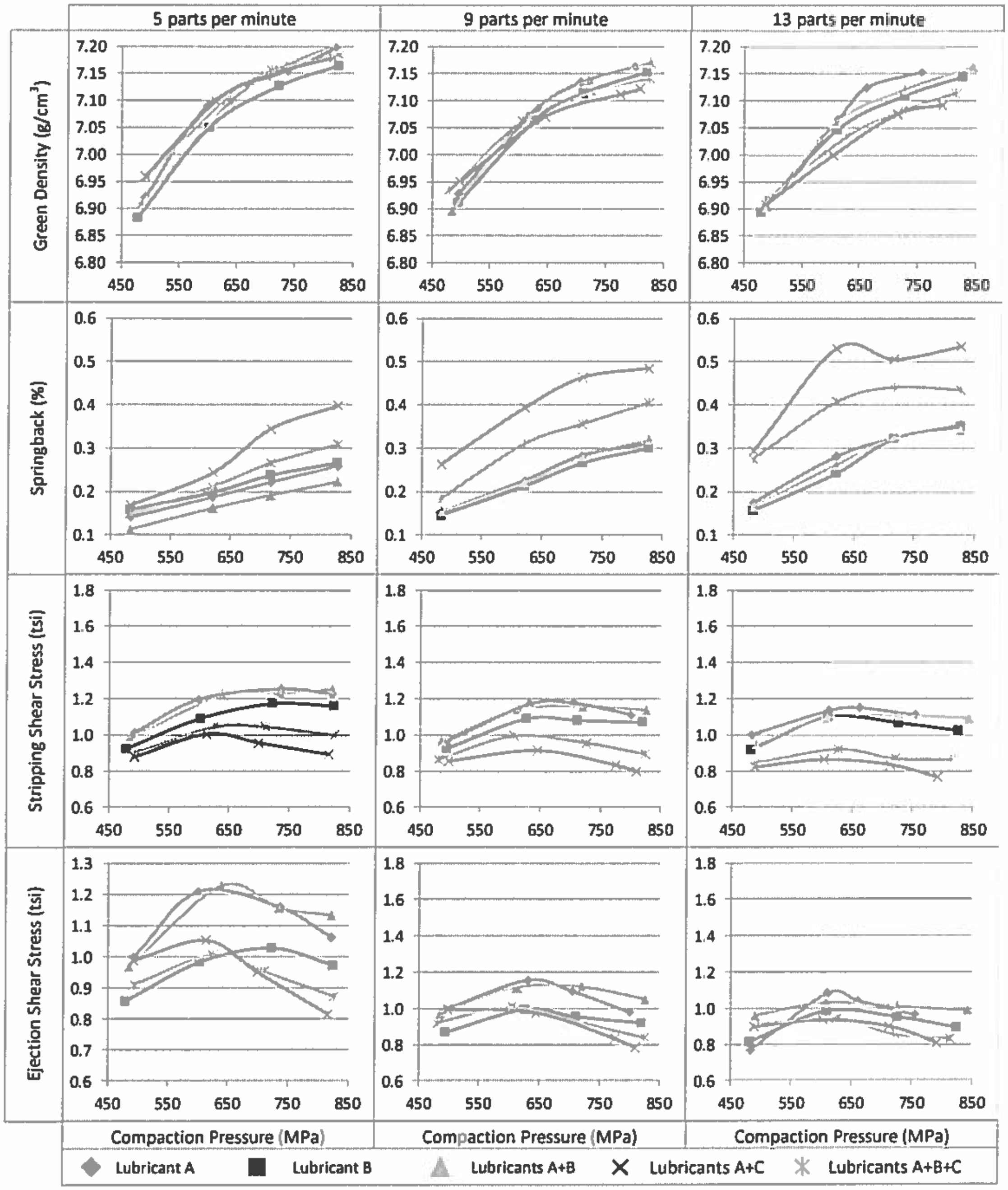

Figure 2. Results of the compaction performed with the heated die (60°C).

Warm Die Compaction (60°C)

Figure 2 shows the exact same data but for these results, the compaction was performed with the die heated to 60°C (warm die compaction technique or WDC). The benefits on the compressibility of WDC technique were notable for the mixes containing EBS. The maximum green density obtained (at higher compaction pressures) increased by about 0.03 g/cm^3. This was not the case for the powder mixes containing low melting point products. For high compaction speeds, mixes 4 and 5 were less compressible than EBS. This reduction in the green density can be attributed to the higher springback for these lubricants. Contrary to the compaction at room temperature, the presence of low melting point products is promoting higher springback which was particularly visible at higher compaction pressures and at high stroke rates. At the fastest stroke rate and the highest compaction pressure, the springback was 26% higher for mix 5 and 56% higher for mix 4.

The ejection performance is also impacted by the use of warm die compaction. Generally speaking, the compaction at elevated temperature contributed to a reduction of the stripping shear stresses of about 14%. Low melting point lubricants also improved the ejection behaviour compared to EBS wax. Akin to the room temperature compaction, an increase in the stroke rate caused a reduction in the stripping shear stresses.

The combination of warm die compaction and the use of low melting point lubricants had a significant effect on the ejection shear stresses. The lowest values were obtained with the combination of WDC, low melting point lubricants and high stroke rates. Once more, the increase in the stroke rate caused a general improvement of the ejection behaviour.

Discussion

Contrary to what has been noted in the literature [5-7], the increase in the compaction rate – and by extension faster punch displacements – lead to reduced ejection forces. This can be tentatively explained by either one of the three following phenomena. Firstly, the compaction process generates a significant amount of thermal energy which needs to dissipate both within the powder volume (including the lubricant) and the die material. With high compaction speeds, this thermal energy has less time to dissipate through the die, due to a shorter dwell time before ejection begins and can lead to increased softening and melting of the lubricants contained in the powder [10]. This softening or melting reduces the viscosity of the lubricant which can then travel to the die part surface more efficiently, thus improving the ejection behaviour. Secondly, the pressurization of the air contained in the powder can extrude some lubricant to the surface with higher velocity thereby enhancing the lubrication. Those mechanisms were not evaluated with precision in this study and would benefit from a more comprehensive study of the compaction mechanisms. Alternatively, at higher compaction speeds, more plastic deformation is achieved which results in an increased surface area of metal powder particles in contact with the die surface. Thus, as the force exerted by the compact on the die is spread over a larger area, the local pressure is proportionally reduced [11].

In all cases, as the compaction speed is increased, there is a net negative impact on the achievable green densities. Two hypotheses could explain this phenomenon. Firstly, as the entire compaction cycle occurs faster, there is less time available for the fine powder particles to rearrange in the voids created between larger particles. This would induce high residual elastic stresses in the small particles which are later released when the compact is freed from the die [12]. The second hypothesis relates to the potential melting of the lubricant which could cause air entrapment.

The positive effect of the larger lubricant particle size could be explained by a volume constraint effect. It is assumed that small lubricant particles can easily travel to voids between the powder particles where they can remain shielded from the inter-particles collisions and particle rearrangement [12]. Such lubricant particles would thus provide little lubricating effect for the ejection process because they would tend to remain located at the core of the compact. Larger lubricant particles, by opposition, would be forced to deform and be expelled to the surface of the compact, in order to form a lubricating film between the part and the die surface. This process remains beneficial as long as the lubricant does not degrade or melt under the heat generated. While it is conceivable that a liquid lubricant would tend to migrate faster toward the die surface, this is not necessarily so as previous research link the migration of the lubricant to the densification process [3]. Nevertheless, assuming that the lubricant migrates due to its reduced viscosity beyond its melting point, the benefits of this phenomenon can still be questioned. As the porosity is rapidly closing under the compaction and deformation of the compact, a significant volume of air needs to escape the powder. It is a possibility that a liquid lubricant would tend to seal off the porosity channels – if not completely, at least partially –

needed for the air to escape. This would lead to air entrapment within the part. As long as the pressure is maintained on the compact, the compressed air is not a problem. Upon the part exiting the die, the now pressurized air would cause some level of elastic relaxation beyond the intrinsic elastic deformation of the material. The observable consequence of the phenomenon is an increased springback. This would also explain why powder mixes containing low melting point lubricants appear to be less compressible. While these powder mixes can reach a high in-die density, the elastic relaxation following the ejection out of the stiff die reduces the final green density.

Conclusion

The use of lubricants with larger particle sizes can contribute to lower the ejection forces due to a better migration of the lubricant to the die to part interface, where its effects are the most desirable. This effect appears to be facilitated by using low melting point lubricants that will soften or partially liquefy by the heat generated through the compaction phase of the part fabrication. However, there seems to be a practical limit to this characteristic of low melting point lubricants. As the temperature is increased, it is possible to melt the lubricant to the extent where some porosity channels – escape paths for compressed air – are being effectively sealed off, causing an internal pressurization of the air trapped in the part. Upon the release of the constraint of the compacted part, elastic relaxation causes the part to swell thereby reducing the green density of the compact.

Differences between various types and particle size distributions of the lubricant tend to be fairly visible at low compaction rates and low temperatures. However, as the compaction speeds and temperature are increased, those differences become less obvious. While no clear explanation was found to explain the observed behaviour of those lubricants, further investigation remains important as industrial compaction of metal powders is regularly done under fast stroke rates and at elevated temperatures.

References

1. Beiss, P., *Technical and Theoretical Aspects of Pressing.* PM98: 1998 Powder Metallurgy World Congress & Exhibition, 1998.
2. http://www.imfine.ca/documents/10180/12243/DWLS+Brochure
3. Gagné, M. and Y. Thomas, *Effect of compaction temperature on the lubricant distribution in powder metal parts*, in *1998 International Conference and Exhibition on Powder Metallurgy and Particulate Materials*. 1998, Metal Powder Industries Federation: Las Vegas, NV (USA). p. 11.39-11.53.
4. Hjortsberg, E., L. Nyborg, and H. Vidarsson, *Microscopic characterisation of topography and lubricant distribution on surface of powder compacts.* Powder Metallurgy, 2005. **48**(4): p. 345-353.
5. Sun, C.C., *Dependence of ejection force on tableting speed—A compaction simulation study.* Powder Technology, 2015. **279**(0): p. 123-126.
6. Mallender, R.F., C.J. Dangerfield, and D.S. Coleman, *The variation of coefficient of friction with temperature and compaction variables for iron powder stearate-lubricated systems.* Powder Metallurgy, 1974. **17**(34): p. 288-301.
7. Bonnefoy, V., P. Doremus, and G. Puente, *Investigations on friction behaviour of treated and coated tools with poorly lubricated powder mixes.* Powder Metallurgy, 2003. **46**: p. 224-228.
8. Thomas, Y., et al., *Effect of lubricant particle size distribution on the processing and properties of PM ferrous parts*, in *EPMA 2013*. 2013, European Powder Metallurgy Association: Göteborg (Sweden). **2**: p. 157-162.
9. Lawrence, A.I., S.H. Luk, and J.A. Hamill, *A performance comparison of current P/M lubricants and routes to improvement*, in *International Conference on Powder Metallurgy & Particulate Materials*. 1997, Metal Powder Industries Federation: Chicago, IL (USA).
10. Zavaliangos, A., et al., *Temperature evolution during compaction of pharmaceutical powders.* Journal of Pharmaceutical Sciences, 2008. **97**(8): p. 3291-3304.
11. Turenne, S., C. Godere, and Y. Thomas, *Effect of temperature on the behaviour of lubricants during powder compaction.* Powder Metallurgy, 2000. **43**: p. 139-142.
12. Koynov, A., F. Romanski, and A.M. Cuitiño, *Effects of particle size disparity on the compaction behavior of binary mixtures of pharmaceutical powders.* Powder Technology, 2013. **236**(0): p. 5-11.