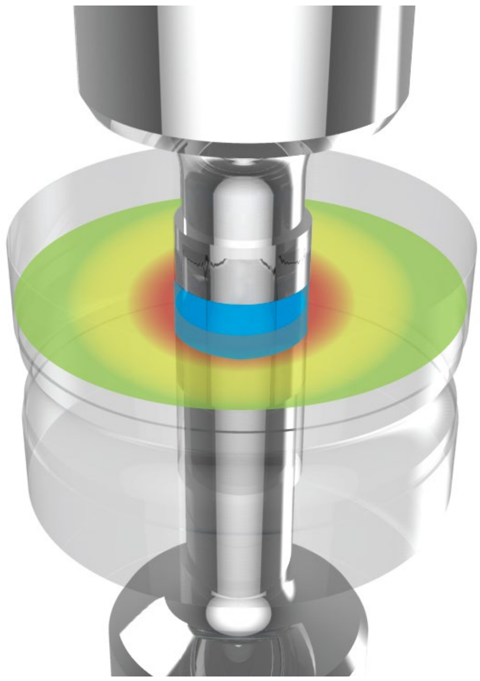
RADIAL DIE WALL PRESSURE MEASUREMENT

Instrumented die is used to measure maximum die-wall pressure (MDP) and residual die-wall pressure (RDP) during powder compaction. This document aims to provide a methodology to characterize powders with this device.



Introduction

During tablet development, people study ejection forces (Fej) in order to avoid tablet defects during tablet manufacturing. Some of those defects, like lamination and capping are said to be linked to high ejection forces. High ejection forces can be induced by high die-wall force or friction between powder and die-wall (Frad =Force radial). To determine the origin of this phenomenon it can be useful to measure radial pressure. Indeed standard compaction studies are made by analysis of the vertical forces on upper and lower punches. However, the die also applies its own pressure to the powder horizontally. This strain, called the die wall pressure, is said to be the hidden variable of the compression.

The pressure of the powder to the die (Radial pressure) is computed by dividing the force by this surface area).

Installing an instrumented die on your STYL'One allows you to measure the die-wall pressure and to perform an extensive characterization of your products. The instrumented die can be used to measure Maximum Die-wall Pressure (MDP) (at the compression peak) and Residual Die-wall Pressure (RDP) (when the axial pressure is removed).

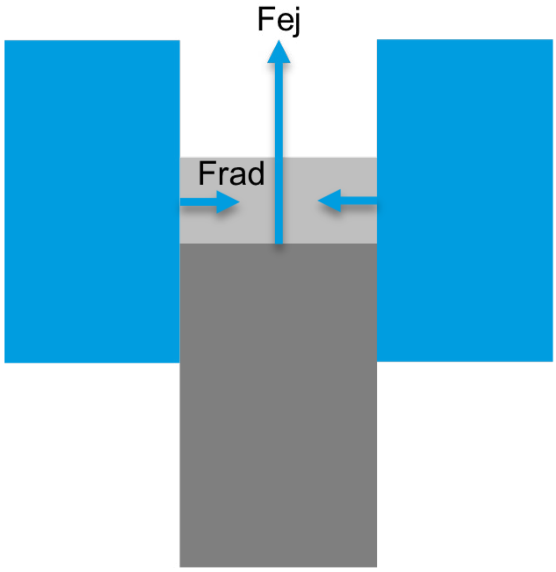


Figure 1
Influence of radial forces on ejection forces [Mazel – Instrumentation and radial pressure].

Instrumentation

As a standard, the instrumented die is plug-and-play device mounted on an 11.28 mm diameter round die with its own set of punches. The instrumented-die is an accessory of STYL'One.

The calculation of the die wall pressure uses the die-wall signal (radial) and the surface area between the compact and the die.

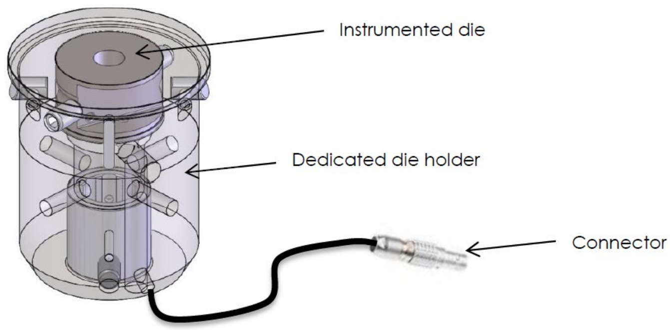


Figure 2
Instrumented die drawing.

$$\text{Die-wall pressure (MPa)} = \frac{\text{Radial force (kN)}}{\text{Surface area (cm^2)}}$$

The surface is automatically computed by the software, by taking into account the thickness (distance between the punches) and the perimeter of the die.

METHODOLOGY

The medium axis of the compression zone is ideally set at 6mm, where the strain gage is located but different depths of compression can be used and the Analis software will perform the correction thanks to the abacus generated during the calibration.

The instrumented die can be used to measure die-wall pressure up to 300MPa. Above some damage can occur to the die, so a safety threshold exists on the software to stop the machine if the stress reaches this limit.

To compare products, a compression speed, a compression pressure and a corrected thickness have to be fixed. At MEDELPHARM Science Lab, a 200MPa axial pressure and a 3mm corrected thickness are targeted. As a consequence, the filling height has to be adjusted step by step to meet those targets. Once the settings are found, two others replicates are performed in order to avoid outliners. For characterization purpose the V-Shape cycle has to be used. The first measurements can be performed at low speed (3mm/s) and some can be performed at high speed (300mm/s). Indeed the speed might have an influence on the deformation behavior.

RESULTS

The whole evolution of the die wall pressure can be easily displayed. The Maximum Die-wall Pressure (MDP) and the Residual Die-wall pressure (RDP) are given by the Analis software. You can even draw plots of MDP and RDP as a function of the mean compression pressure.

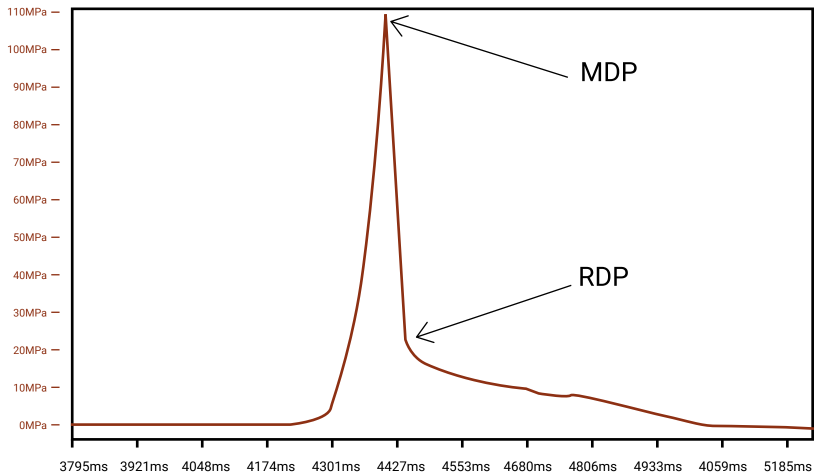


Figure 3
Radial pressure signal on Analis display tab.

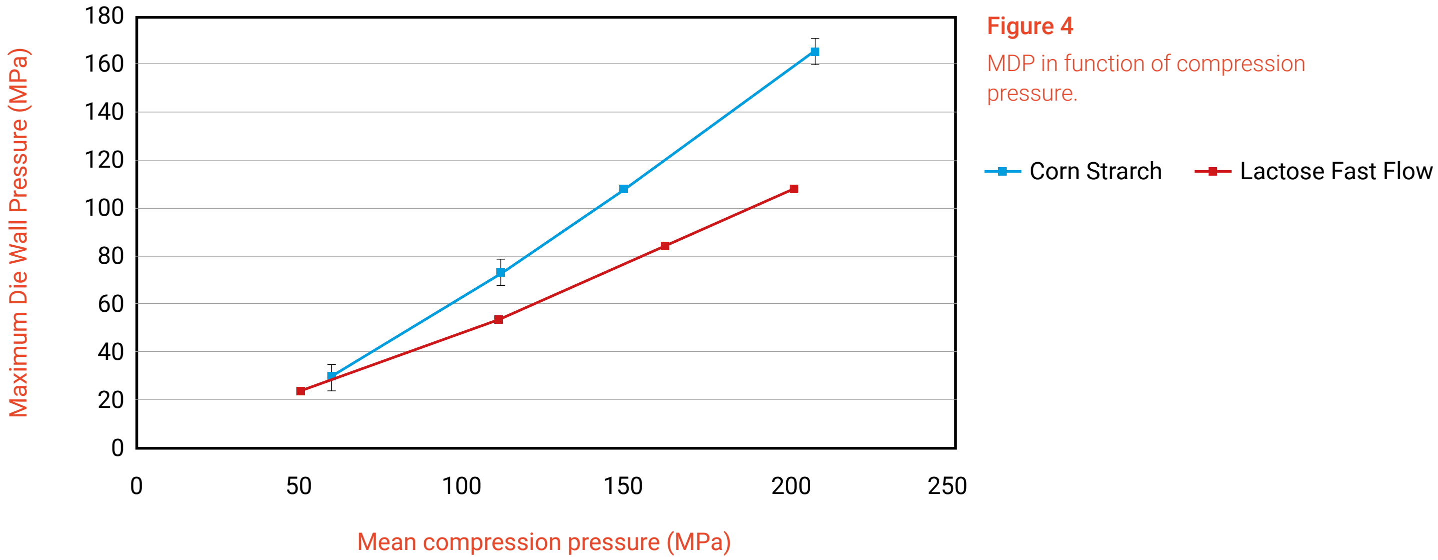


Figure 4
MDP in function of compression pressure.

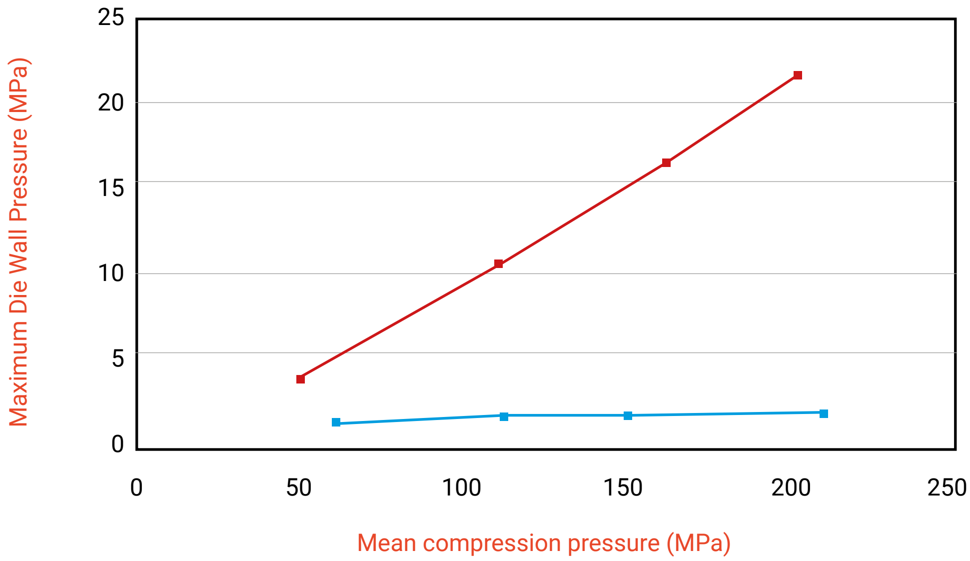


Figure 5

RDP in function of compression pressure.

Corn Strarch
Lactose Fast Flow

Graphical representations of MDP and RDP of corn starch and lactose in function of compression pressure indicate a higher MDP and a lower RDP for corn starch than for lactose.

DISCUSSION – CONCLUSION

Corn starch well-known to be a plastic/elastic product presents a higher MDP than lactose (plastic/brittle product), but a lower RDP.

Same observations were presented by Abdel-Hamid & Betz in 2011. In his study, it is said that an increasing RDP value during compaction would indicate higher tendency for friction, whereas an increased or constant value of MDP would provide an evidence for plastic behavior, a character always desired in powder formulations.

RDP can also be used to predict capping propensity but it seems to be product dependent. According to Mazel et al. (2015) high RDP can lead to capping, linked to higher tendency for friction.

Paul & Sun (2017) have shown that capping positively correlates with low RDP. Indeed, product with elastic behavior display low RDP, and high elastic recovery can lead to capping.

The MEDELPHARM Science Lab can help you developing or improving your formulation with STYL'One compaction simulator. Also solving capping issue can be easily made by determining MDP and RDP of your formulations using the instrumented die.

STYL'One Evo key benefits

- Versatile
- Standard tooling
- Ideal for small amount of material
- Quick product and tooling changeover
- Easy to clean – easy to handle
- Simulation of any rotary tablet press
- User-friendly HMI for fast experiment setup and results with automatic studies

References:

Abdel-Hamid, S. & Betz, G. Study of radial die-wall pressure changes during pharmaceutical powder compaction. Drug Development and Industrial Pharmacy 37, 387–395 (2011).

Mazel, V. et al. Evolution of the Die-Wall Pressure during the Compression of Biconvex Tablets: Experimental Results and Comparison with FEM Simulation. Journal of Pharmaceutical Sciences 104, 4339–4344 (2015).

Paul, S. & Sun, C. C. Gaining insight into tablet capping tendency from compaction simulation. International Journal of Pharmaceutics 524, 111–120 (2017)

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