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STRESS STATE OF PHARMACEUTICAL POWDER DURING UNIAXIAL COMPRESSION

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Abstract: Nowadays tabletting of powders in the pharmaceutical industry is, among other agglomeration processes, most widely used method of finalizing solid products. From a procedural point of view, stress fields of the tablet have the dominant importance because they directly determine its quality. This paper focuses on the experimental detection of the primary stresses during the compression of the pharmaceutical powdered substance AVICELL. Measurements will be performed on the KISTLER programmable electromechanical press in a die with a circular cross-section. Evaluation of the obtained data of the stress fields will be carried out by means of Mohr's circle.
Keywords: uniaxial compression, pharmaceutical powder, stress state, Mohr´s circle

INTRODUCTION

Tablet production by means of uniaxial compression is the dominant processing technology of powder materials. Among the areas of application of the technology belong powder metallurgy, filters, magnets and electrical contacts. Uniaxial compression plays an irreplaceable role in the pharmaceutical industry. The advantages of this technology are cost-efficiency, high productivity and low waste production.

THEORY

Analysis of the mechanical properties of the powder during compressing

The analysis of mechanical properties is focused on monitoring force action inducing change in the bulk density of the powder. These parameters are most often expressed as stresses and strains and their relation to the particular materials is a frequent subject of investigation. Based on the mechanics of solids, the principles of this investigation are implemented into the theory of compaction of powder materials.

Distribution of stresses during compression

During compression, the piston acts on the powder by compression force and creates a stress state in the powder. Due to fact that the powder is not a continuum, axial stress is not constant, but decreases with increasing distance from the pressing piston. Part of the axial load is converted to a radial which acts on the die wall, creating friction between the wall and the powder. The situation is shown in Figure 1, when the powder is compressed in a cylindrical die diameter D. Consider a cylindrical tablet of diameter D and height H and an elemental slice dz situated at distance z from the bottom of the tablet. Neglecting the weight of the powder, the force balance equation is:

$$\frac{\pi D^2}{4} d\sigma_z = \tau_z \pi D dz \qquad (1)$$

where $d\sigma_z$ is the elementary increase of axial stress, τ_z is shear stress, dz is elementary increase of axial position. The radial stress at the given height z can be written as:

$$\sigma_r = K\sigma_z \qquad (2)$$

where σ_r is radial stress at axial position z, σ_z is axial stress at axial position z and K is radial-to-axial stress ratio. The shear stress due to the friction acting on the elemental slice can be expressed according to Coulomb's law of friction:

$$\tau_z = \mu\sigma_r \qquad (3)$$

where μ is the friction coefficient. Combining equations (2) and (3), the equilibrium equation becomes:

$$\frac{d\sigma_z}{\sigma_z} = \frac{4K\mu}{D} dz \qquad (4)$$

If it is assumed that the product Kμ is independent of position z, equation (4) can be integrated using the appropriate boundary conditions:

$$\ln\frac{\sigma_z}{\sigma_B} = \frac{4K\mu}{D}z \qquad (5)$$

or

$$\ln\frac{\sigma_T}{\sigma_B} = \frac{4K\mu}{D}H \qquad (6)$$

Equations (5) and (6) imply the exponential distribution of the axial stress at a given height in the tablet:

$$\sigma_z = \sigma_B \exp\left(\frac{4K\mu}{D}z\right) \qquad (7)$$

or

$$\sigma_z = \sigma_T^{\frac{z}{H}} \sigma_B^{\left(1-\frac{z}{H}\right)} \qquad (8)$$

If the values of the stresses acting on the top and bottom piston are known, it is possible to determine the value of the axial stress at each point of the tablet on the basis of equation (8). If the radial stress is measured at one position along the tablet height, equation (8) determines the axial stress at the given position. Then it is possible to determine the stress state in the powder using a pair of principal stresses σ_1 and σ_2, where:

$$\sigma_1 = \sigma_z \qquad (9)$$

and

$$\sigma_2 = \sigma_r \qquad (10)$$

These stresses can be evaluated using Mohr's circle in τ - σ diagram. The diagram expresses the dependence between first and second principal stress and shear stress τ:

$$\tau = \frac{(\sigma_2 - \sigma_1)}{2} \qquad (11)$$

The tangent line for each Mohr's circle is called the yield limit and expresses the flowabillity of a particulate material during compaction.

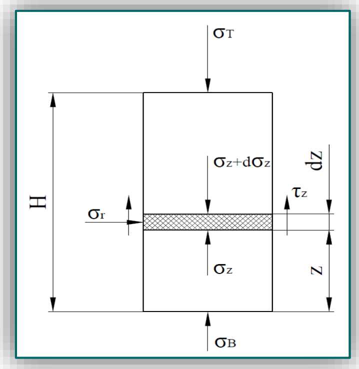

Figure 1: Stresses applied to a powder slice

EXPERIMENTAL STATION

Measurement was carried out on the electromechanical press KISTLER (Figure 2). The press contains a force sensor and a piston rod position sensor. The maximum compression force is 60 kN and the stroke of the piston rod is 200 mm. Pressure control and evaluation of the measured data is carried out by means of the NC module DMF-P A310, thus enabling evaluation of the force in dependence on position.

Figure 2: Electromechanical press KISTLER

EXPERIMENTAL MATERIAL

The material used in measurements was Avicel PH 102. This material is one of the oldest types of microcellulose and one of the most widely used pharmaceutical excipients. Cellulose is used in the powder, and MMC in the microcrystalline form. Its properties make it ideal for the formulation of pharmaceutical forms with a controlled release of the drug, ensuring a prolonged, delayed or pulsed effect of the drug substance in a living organism.

Figure 3: Avicel PH 102

Table 2: Mechanical and physical properties of Avicel

Particle size [μm]	180
Bulk density [kg/m³]	300
Density of solid particles [kg/m³]	1590
Angle of internal friction [°]	34,5
Young's modulus [MPa]	1209
Poisson's ratio [1]	0,148

EXPERIMENTAL MEASUREMENT

The measurement carried out on the equipment allows recording the axial force on the bottom piston and the radial force on the die wall. The bottom piston was static and compression was carried out through the movement of the top piston, which was pushed by an electromechanical press. The press recorded the compression force and piston position. The range of the compression forces was from 10 kN to 50 kN, and the compression speed was 1 mm/s. Force values were converted to stresses with respect to the piston cross-section. The current relative density was calculated on the basis of the measured values of absolute heights of the tablet and known powder mass. The relative density of the tablet is defined as the ratio between bulk density of powder ρ_B and density of particles ρ_T:

$$RD = \frac{\rho_B}{\rho_T} \qquad (12)$$

where the bulk density of the powder is defined by dividing the mass of the powder m and its volume V_B:

$$\rho_B = \frac{m}{V_B} \qquad (13)$$

THE VALUES OF STRESSES DURING UNIAXIAL COMPRESSION OF THE POWDER

The measured values express the axial stresses acting on the top and bottom pistons, the radial stress acting on the die wall and the relative density of the tablets during compression. With respect to equation (8), the value of the axial stress at the point at which the radial stress is measured is calculated. In Figure 4 the evolution of the axial and radial stresses during compaction is shown as a function of the relative density. Pairs of the principal stresses at the radial sensor position are shown in τ - σ diagram as Mohr's circles and appropriate yield limit.

Table 2: Table of measured and calculated values of stresses and relative densities

Relative density RD [1]	Axial stress (top) σ_T [MPa]	Axial stress (bottom) σ_B [MPa]	Axial stress (sensor) σ_z [MPa]	Radial stress (sensor) σ_r [MPa]
0,270	0,000	0,000	0,000	0,000
0,607	31,545	23,344	25,401	1,165
0,713	57,361	43,182	47,415	1,474
0,774	80,726	58,808	65,863	1,993
0,835	115,900	76,862	90,047	4,970
0,910	159,096	102,644	123,398	11,105

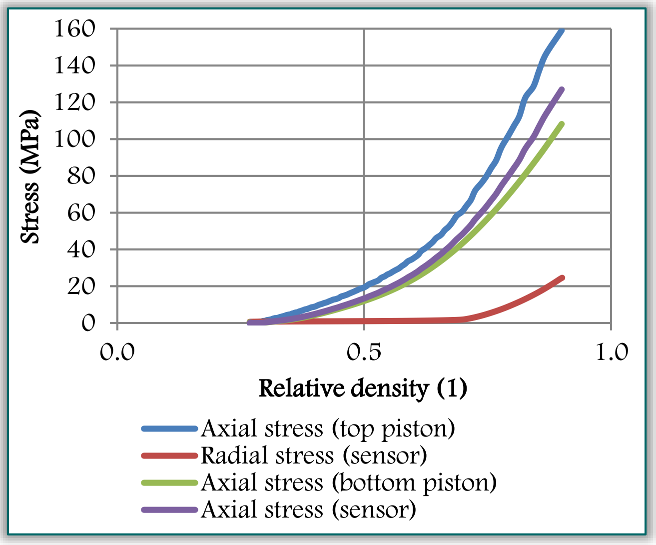

Figure 4: Evolution of the axial and radial stresses during compaction

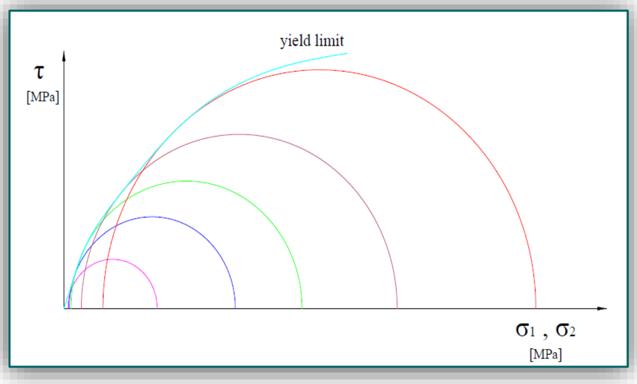

Figure 5: Mohr's circles and yield limit

CONCLUSION

The paper is focused on testing a new measuring procedure for obtaining the data needed to assess the mechanical properties of powders during uniaxial compression. The measurement was carried out on a single-action electromechanical press designed to obtain the laboratory data applicable to production equipment. The stress state arising during powder compaction affects the final quality parameters of the tablet. This data is also important for the design of pressing equipment and tools in industry.

Nowadays the pharmaceutical industry often uses computer simulations to describe and to create the possibility of improving the compression process. In order to carry out such a simulation, it is necessary to first input the mechanical properties of the powder into the simulation program. By the procedure described in this paper can be obtained the development of the axial and radial stresses during compression of the powder, which is also required as input data in the computer simulations.

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Note

This paper is based on the paper presented at The 9th International Conference for Young Researchers and PhD Students - ERIN 2015, May 4-6, 2015, Monínec, Czech Republic, referred here as[9].

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