Some aspects of granulated ceramic powder die compaction

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Abstract. Manufacturing of ceramic goods most often involves compaction of granulated powder. There are numerous phenomena that complicate rheological equations of densification during the compaction process and therefore render these equations practically useless without computer assist. We present a simple generalized model of densification of granulated ceramic powder based on Ballhausen-Gąsiorek’s, Messing’s and Pascal’s models. Experiments have proven a linear variation of density along the direction of compression and changes of dynamic and kinematical coefficients of friction. The variation of the dynamic coefficient of friction is in the range from 0.15 to 0.25.

1. Introduction
Compaction in rigid or semi-rigid moulds is one of the most widespread methods of forming ceramic products from granulated masses. Although it is a dynamic process, it is usually described as a static one, providing that a general rheological equation of state is satisfied, i.e.:

\[ T_n = (D, t) \]  
(1.1)

where:
- \( T_n \) - tensor of stresses [Pa] 
- \( D \) - tensor of deformations [m] 
- \( t \) - time [s]

and scalar equation (common name compaction equation):

\[ \rho = (P, t) \]  
(1.2)

where:
- \( \rho \) - density [g/cm³] 
- \( P \) - pressure [Pa] 
- \( t \) - time [s]

or combination of this.

Classical compaction (commonly called uniaxial) can be one-sided, two-sided, or pseudo-two-sided with regard to the applied compression direction [1]. Numerous equations that describe the relation between compact density (also volume or porosity) and the applied compression pressure are reported in the literature [2-16].

These equations assume that the coefficient of friction between ceramic granulated powder and a die-wall does not change. However friction forces between granules and die-wall during pressing lead to unequal distribution of compact density. It is known, from the view point of physics, that materials that have different densities also have different coefficients of friction. It should be emphasized that
the purpose of densification is to change density. This research shows that the changes in friction factor constitute a function of density and corroborate the following theories: the Jansen-Walker Cap (DPC), the critical states conceptions, and other elastic-plastic models [17-23]. However, in the above-mentioned conceptions, the friction factor variations depend on other factors, e.g. adhesion, material elasticity, etc. On the other hand, theories foreseeing the friction factor variations in a function of the consolidation degree, especially those which use the finite element method, arbitrarily assign a few values of it within 0<m<1, while analyzing the density distribution inside the compact afterwards up to Pascal’s law (i.e. 0-solid body, 1-liquid body) [23].

The purpose of a suitable selection of mathematical transformations of densification equations is to obtain a linear description and determination of directional coefficients, i.e. material constants. The models of Thompson or Ballhausen modified by Gąsiorek [2, 5, 9] are also used to describe stress distribution within compact.

Thompson's model relates to the uniaxial two-sided compaction, nevertheless, it is mathematically complicated and difficult to use in practice. Moreover, it requires many mutually entangled material constants. On the other hand, Ballhausen-Gąsiorek’s model lends itself more easily to practical applications but it refers only to the uniaxial one-sided compaction.

Experiments showed that average density of compaction along the direction of compression changes exactly linearly. The linear variation of density along the densification direction have been confirmed also by ultrasound experiments at frequency of 0.5 MHz (Figure 1).

It should be noted that the linear change of bulk density along the direction of compression can be neither applies to the central axis, nor to the walls area – it represents the average density of complicated circular shapes of the layers (pallets).

![Figure 1. Changes of waves propagation speed at frequency 0.5 MHz vs. density of layers](image)

2. Experimental
Our laboratory experiments were conducted using the equipment specially designed for this purpose. Its scheme is shown in Figure 2.
Figure 2. Schematic diagram of test rig: 1) DC motor, 2) belt gear, 3) worm gear, 4) flexible clutch, 5) steel plate, 6) punch, 7) leading punch, 8) die, 9) hydraulic servo-motor, 10) hydraulic system, 11) torque measuring path, 12) pressure force measuring path, 13) strain gauge amplifier, 14) recorder

The measurements were completed within the same pressure range of 40-100 MPa as during densification. The sliding speed of the sample on the steel plate was 0.02 m/s. The plate, made from thermally hardened NC4 steel of 55HRC hardness, was part of the friction measurement equipment. A specimen of the ceramic granulated material was placed in a cylindrical power bed 20 mm in diameter and compacted by the upper punch. The volume of the specimen was set experimentally (height of 2/3 of diameter) in order to obtain high degree of densification.

3. Results and discussion

3.1. Model of granulated ceramic powder densification

Qualitative and quantitative changes of static and kinetic coefficients of friction between granulate and steel are shown in Figure 3.
It can be seen that both coefficients of friction vary with the change of compaction pressure. In particular, during compaction in the range of 40–100 MPa, the kinetic coefficient of friction clearly changes its value. It can be assumed, not considering the effective transfer of normal stresses from the upper punch through specimen to lower steel plate, that a different level of mass density is obtained in the place of contact for various pressures. If this is the case, then the masses of different density levels have different coefficients of friction what was assumed by the described generalized model of ceramic powders densification.

Based on the results, we offer a modified simple model compaction (Figure 4). We start with the assumption that the compaction force consists of a vertical component (normal stress) and a horizontal one (tangential stress). Normal stress is responsible for compact density while tangential stress creates die-wall friction and is responsible for uneven distribution of powder density. Following Pascal’s law the tangent of the angle of distribution of these forces equals 1 for fluid and 0 for a rigid body. It is also assumed that both stresses are the function of compact height. It should be noted that one of the purposes of ceramic powder granulation is to make a ceramic mass more susceptible to the process of formation in order to ease the form fill up and make it more even.

\[ \delta(h) = \frac{P(h)}{F} \text{ and } \tau(h) = \delta(h)\tan[\varphi(h)] \]  
\[ \frac{dN(h)}{dh} = \tau(h)\delta(h) \]  
\[ dP(h) = \mu(h)dN(h) \]  
\[ dP(h) = \frac{S}{F} P(h)\mu(h)\tan[\varphi(h)]dh = \frac{S}{F} C(h)P(h)dh \]  
\[ \frac{dP}{P} = \frac{dP(h)}{P(h)} = \frac{S}{F} C(h)dh \]

For reduction mathematical formula assuming that \( C(h) = \mu(h)\tan[\varphi(h)] \) we obtain:
\[
\ln P(h) - \ln P(0) = \frac{1}{P} \int_0^h \frac{S}{F} C(h) dh = \frac{1}{F} \int_0^h C(h) dh (3.6)
\]

From here:
\[
\ln P(h) = \frac{S}{F} \int_0^h C(h) dh + \ln P(0) (3.7)
\]

and:
\[
P(h) = \frac{S}{F} \int_0^h C(h) dh + \ln P(0) (3.8)
\]

where:
\(\delta\) - normal stress [Pa],
\(\tau\) - shear stress [Pa],
\(P\) - pressure [Pa],
\(F\) - section field \([m^2]\),
\(\phi\) - distribution angle of unit pressure \([\circ]\),
\(h\) - height (counted from central cross section) \([m]\),
\(dN(h)\) - pressure on side \(dh\) on heights \(h\), (increment of cross stress) [Pa],
\(dP(h)\) - increment of pressure [Pa],
\(\mu(h)\) - coefficient of friction between powder and die-wall [-].
\(S\) - circumference of compaction \([m]\).

Equation (3.8) is a generalized formula for stress distribution within the entire volume. Similar expressions were derived earlier, assuming a constant value of the friction coefficient[18-23].

The research has shown [1, 20, 21] that the following equation is useful for the description of the powder densification process:

\[
\rho_w(h) = A \ln[P(h)] + B (3.9)
\]

Referring to previous assumptions with regard to linear stress distribution along compression direction we have:

\[
\rho_w(h) = Kh + L (3.10)
\]

where:
\(\rho_w\) - relative density \([kg/m^3]\),
\(A, B, K\) and \(L\) - constants [-],
\(h\) - height \([m]\).

Comparing equations (3.9) and (3.10) we obtain:

\[
Kh = A \ln[P(h)] + B - L = A \ln[P(0)] + \frac{S}{F} \int_0^h C(h) dh + B - L = \frac{S}{F} \int_0^h C(h) dh + A \ln[P(0)] + B - L (3.11)
\]

Differentiating above \((h\) is variable) we obtain:

\[
K = \frac{S}{F} C(h) \text{ i.e. } C(h) = \frac{F}{S} = \text{const} (3.12)
\]

thus:

\[
C(h) = \mu(h) \tan[\phi(h)] = \text{const} (3.13)
\]
This expression determines that $C(h)$, i.e. the product of the coefficient of friction and the tangent of the angle of stress distribution, is constant along the direction of compression (material constant). It means that the coefficient of friction may change along the compression direction if there is a change in the stress distribution angle as shown in Fig. 3. A similar result was obtained by Briscoe and Rough [22]. Also Michrafy et al. [23], who considered Walker’s elasticity coefficient because they had assumed, similarly to others, a constant value of the die-wall coefficient of friction.

Considering equation (3.8), it is possible to determine the influence of granulated mass on densification, i.e. critical pressure of the granules destruction and plastic flow formation [24-32]. Plastic flow is the main mechanism in the consolidation of ceramic powders. In the multistage consolidation of ceramic granules with pressure, the elastic deformation analysis is not well-founded but particularly when referring to ceramic powders which do not contain organic binders (Figure 5).

According to this model, in the range of plastic flow, the compaction density is only a function of pressure ($P$) and three material constants: (A) coefficient of densification, (B) bulk density of granulated ceramic mass and (C) coefficient of skid, i.e. a product of the coefficient of friction and the tangent of the angle of stress distribution. In order to validate this model, the following elements should be proved: (1) linearity of bulk density along compression direction and (2) variation of coefficient of friction on die wall.

4. Conclusions
1. The product of the coefficient of friction and the angle of stress distribution along the direction of pressing are constant.
2. There are variations in the friction factor between the ceramic powder and the die wall during the consolidation.

References
[1] Izak P 2002 Polish Ceramic Bulletin 70 5 (in Polish)
[2] Izak P, Stochel J 2000 Ceramika 60 157 (in Polish)
[3] Gąsiorek S, Maciejko K, Szatkowska J 1980 Materials Science Monographs 6 223
[4] Gąsiorek S 1979 Polish Ceramic Bulletin 40 41 (in Polish)
[5] Patel S S, Patel N M 2009 International Journal of Pharmaceutical Research 1 2
[6] Hyoung S K, Sung-Tag O, Jai-Sung L 2002 Journal of the American Ceramic Society 85 2137
[7] Balakrishnan A, Martin C L, Bhasker P S, Shrikant J 2011 Journal of the American Ceramic Society 94 (4) 1046
[8] Thompson R A 1981 Ceramic Society Bulletin 2 237
[9] Groenou V, Broese A 1981 Powder Technology 28 221
[10] Azhdar B, Stenberg B, Kari L 2006 Polymer Testing 25 114
[11] Frenning G 2007 Powder Technology 172 103
[12] Han L H, Elliott J A et al. 2008 International Journal of Solid and Structures 45 3088
[13] Ballhausen C 1951 Archiv fuer das Eisenhuettenwesen 22 185
[14] Izak P 1993 ECERS –Third Conference P0 4
[15] Izak P 2001 Keramische Zeitschrift 53 912
[16] Messing G L, Onada GY 1978 Journal of the American Ceramic Society 61 1
[17] Messing G L, Markhoff C J, McCoy L G 1982 Ceramic Society Bulletin 61 857
[18] Lukasiewicz S J, Reed J S 1978 Ceramic Society Bulletin 57 798
[19] Sastry K V S, Fuerstenau D W 1973 Powder Technology 7 97
[20] Aydin I, Briscoe B, Sanhturk K Y 1996 Powder Technology 89 239
[21] Cameron I M, Gethin D T, Tweed J H 2002 Powder Metallurgy 45 345
[22] Briscoe B J, Rough S L 1998 Colloids Surface A: Physicochemical Engineering Aspects 137 103
[23] Michrafy A, Dodds J A, Kadiri M S 2004 Powder Technology 148 53
[24] Zhang B, Jain M, Zhao C, Bruhns M, Lawcok R, Ly K 2010 Powder Technology 204 27
[25] Sinka I C, Cunningham J C, Zavaliangos A 2003 Powder Technology 133 33
[26] Carnein R D, Messing G L 2001 Powder Technology 115 131
[27] Strijbos S 1977 Powder Technology 18 334
[28] Khoei A R, Keshavarz S, Biabanaki S O R 2010 Finite Elements in Analysis and Design 46 843
[29] Ramakrishnan N, Arunachalam V S 2005 Journal of the American Ceramic Society 76 2745
[30] Ranachowski J, Rejmund F, Librant Z 1985 Archiwum Nauki o Materiaalach 6 211 (in Polish)
[31] Tabata T, Masaki S, Kamata K 1981 Powder Metallurgy International 13 179
[32] Klemm U et al. 1997 Journal of the European Ceramic Society 17 141