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# Particle Size Range as a Factor Influencing Compressibility of Ceramic Powder

**Mirjana Djurić,[a] Radmila Marinković-Nedučin,[a] Jonjaua Ranogajec[a] & Miroslava Radeka[b]**

[a] Faculty of Technology, [b] Faculty of Civil Engineering, University at Novi Sad, 21000 Novi Sad, Serbia

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**Abstract**: The paper reports on an investigation conducted with the aim to determine whether an overall deformation stress of ceramic powder depends on the size of particles present in a system. For this purpose, six fractions (obtained by sieving of raw material, so as to contain particles with defined size range) were compacted and their compaction response diagrams were generated. For each particular fraction the parameters of the Heckel compaction model were calculated and used for estimating the deformation stress of each particular fraction. A correlation between average diameter of particles and the overall deformation stress of powder was found.

## 1 INTRODUCTION

Pressing of ceramic powders in a die has undoubtedly received the attention of scientists for many years, while powder compaction is an operation very often applied in the process of ceramics production. The characteristics of the green article are very important and how much the final quality of the product is affected by them goes without saying.

Many papers deal with the compaction behaviour of powders in terms of applied pressures. Bruch[1] was the first who investigated the consolidation of ceramic powders, while Neisz *et al.*[2] made an effort to explain the fundamental principles.

Behaviour of any powder exposed to the influence of pressure, can be investigated by an analysis of its compaction response diagram which represents relative densities of compact (in terms of density of initial powder) as a function of applied pressures. It is always obtained experimentally, by a standardized procedure, in a compression simulator. Graphical representation of the pressure–density relation has been considered by Lukasiewicz and Reed,[3] Messing *et al.*[4] and by many other authors.[5–7] The mathematical description of compaction can be given by general equations, based on various theoretical or empirical assumptions (see Heckel,[8] Cooper and Eaton,[9] Kawakita and Ludde[10] and James[11]).

Analysis of compaction diagrams leads to the conclusion that various factors play an important role in the compaction process; among others, powder characteristics resulting from the size and the shape of each particle should be especially pointed out. Huffine and Bonilla[12] and Leiser and Whittemore[13] have investigated the influence of particle size, distribution of particle sizes and the shape factor uniformity upon the compressibility of powders.

Here also, the influence of the particle size range on the deformibility of atomized ceramic powder was considered. For this purpose, six fractions were separated from the same industrial batch (by sieving) and their compaction response diagrams were generated. An overall deformation stress for each particular fraction was calculated, by using Heckel's exponential model,[8] and correlated with the average diameter of particles.

## 2 MATHEMATICAL MODEL OF COMPACTION

As for compaction of powders of various inorganic materials, that of ceramic powders can be expected to proceed by one or more models of deformation (elastic, plastic and brittle) which are quantified by adequate deformation stresses.[14]

Apart from elastic deformation (less typical of ceramic powders), there is a general relationship between the two remaining kinds of deformation. It defines the moment (a necessary condition) when the ductile behaviour transforms into the brittle one:

$$\sigma_p = \sigma_b \tag{1}$$

Before this moment, plastic flow (dependent on the yield stress $\sigma_p = \sigma_y$) is dominant, while afterward brittle fracture (dependent on the toughness of material, represented by $K_{IC}$, and the particle size, given by the average diameter) appears. The relation which expresses the brittle deformation stress as a function of both $K_{IC}$ and $d$ has the following form:

$$\sigma_b = A\ K_{IC}/(d)^{1/2} \tag{2}$$

where $A$ is a constant depending on the geometry and loading system.

From this equation it is evident that an increase of particle diameter causes a decrease of the stress required for brittle fracture, which becomes less than that needed for plastic flow. Therefore, cracking of large particles is much easier than cracking of small particles. On the contrary, small particles show plasticity because the plastic deformation stress is lower than the brittle deformation stress. So, eqns (1) and (2) define the correlation between the average particle size and the deformation stresses.

On the other hand, an average deformation stress of a material that is plastically deformed, could be predicted by the modified Heckel equation:[8]

$$\ln[1/(1\text{-}D)] = KP + B = (1/\sigma_p)\ P + B \tag{3}$$

which expresses the relative density of a material ($D$) as a linearized exponential function of the compression pressure, with $K$ and $B$ as the parameters.

The idea that the reciprocal of $K$ can be (numerically) equalized to the mean yield stress was suggested by Hersey and Rees,[15] and approved by Roberts and Rowe,[16] for certain ductile metals and polymers. However, in the case of a material where both plastic deformation and brittle fracture are occurring, eqn (3) should contain $\sigma_d$ which represents the deformation stress of particles, be it a plastic deformation stress, a fracture deformation stress or a combination of the two.

So, a general exponential law of compaction, in its nonlinearized form, can be expressed by the equation:

$$D = 1 - \exp\{-[(1/\sigma_d)\ P + B]\} \tag{4}$$

## 3 EXPERIMENTAL

### 3.1 Ceramic powder

Atomized ceramic powder, used in all experiments, was taken from the industrial raw material batch (for ceramic roof tiles production) and separated by sieving in six fractions of defined particle size range (see Table 1). By employing standard methods, physical and chemical characterization of each particular fraction was performed; so, the surface area was determined by static low-temperature nitrogen adsorption (ASAP 2000, Micromeriotics), while the density of initial powder was determined by a pycnometer. As far as chemical and phase composition are concerned, well known chemical methods and X-ray powder diffraction (Philips PW 1050, $CuK_\alpha$) were applied. The microstructure was investigated by SEM (JOEL JSM 35). Results of these analysis are partly given in Table 1.

### 3.2 Compaction

Compaction of all samples was carried out by Instron-press 1122 (die dimensions: $d$ = 7 mm and $h$ = 5·5 mm ) with a crosshead speed of 50 mm/min in the pressure interval up to 31·5 MPa. Compaction response diagrams were obtained indirectly, by screening the compaction root (crosshead displacement depending on applied force). For a known height and weight of load, and dimensions of the die, the density of the resulting compact was calculated. When this value

**Table 1. Some physical and chemical characteristics of particular fractions**

| Fraction | Particle size $d$ (mm) | Powder density (kg/m$^3$) | Specific surface area (m$^2$/g) | Chemical composition | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | $SiO_2$ (%) | $Al_2O_3$ (%) | $Fe_2O_3$ (%) | CaO (%) | MgO (%) | $Na_2O$ (%) | $K_2O$ (%) |
| 1 | 0·06–0·09 | 3020 | 13·1 | 52·9 | 15·4 | 2·7 | 8·8 | 4·3 | 1·0 | 2·2 |
| 2 | 0·09–0·2 | 2330 | 15·3 | 52·3 | 14·6 | 2·8 | 8·9 | 4·1 | 1·4 | 2·4 |
| 3 | 0·2–0·315 | 2700 | 15·2 | 52·6 | 14·3 | 3·4 | 9·0 | 4·4 | 1·1 | 2·2 |
| 4 | 0·315–0·4 | 2700 | 14·4 | 54·2 | 15·0 | 3·0 | 6·7 | 5·1 | 1·3 | 2·1 |
| 5 | 0·4–0·5 | 2700 | 14·8 | 53·5 | 16·0 | 2·3 | 7·4 | 4·0 | 1·4 | 2·4 |
| 6 | >0·5 | 2700 | 14·5 | 53·7 | 15·0 | 3·1 | 7·1 | 3·9 | 1·5 | 2·3 |

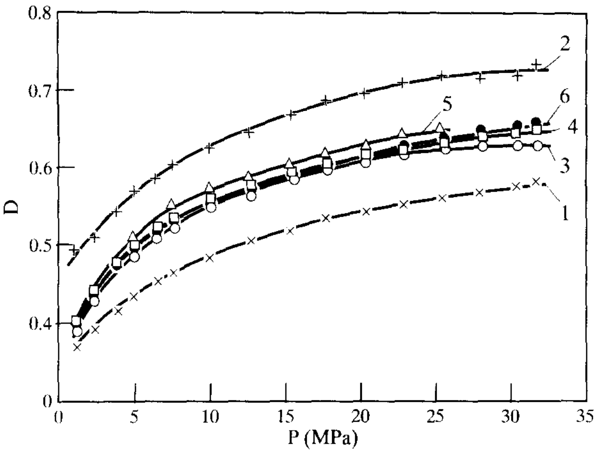


**Fig.1.** Compaction response diagrams for particular fractions.

was divided by the maximal possible density (measured by pycnometry) the relative density of every sample was obtained.

Compaction response diagrams for all investigated fractions (size ranges) are presented in Fig. 1.

## 4 DISCUSSION OF RESULTS

When the Heckel exponential function was adopted as a model of ceramic powder behaviour and the least square method was applied (on the measured data basis) the results presented in Table 2 were obtained. Small relative deviations of measured values from the calculated results (column five in Table 2) show a high degree of the exponential law obedience. So, there is no doubt that eqn (4) can be used for estimation of the overall deformation stress, typical of each particular fraction (see last column in Table 2).

When these values were taken as a basis for rearranging the fractions in a series, so that the deformation stresses formed a parallel series whose terms decreased in magnitude, the order presented in Table 2 was obtained. This order can be used for drawing conclusions concerning the relation between average diameter of particles and the deformation stress of the fraction that contains these particles. In the case of the investigated

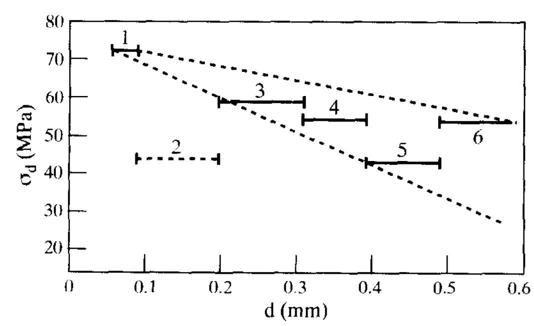


**Fig.2.** Relation between deformation stress and diameter of particles.

**Table 2. Parameters for the Heckel model**

| Fraction | Particle size $d$(mm) | $K(\times 10^2)$ | $B$ | $\delta$ (%) | $(1/K) \equiv \sigma_d$ |
|---|---|---|---|---|---|
| 1 | 0·06–0·09 | 1·3503 | 0.5005 | 2·697 | 74·06 |
| 3 | 0·2–0·315 | 1·6521 | 0·5730 | 4·008 | 60·53 |
| 4 | 0·315–0·4 | 1·7632 | 0·5762 | 3·575 | 56·72 |
| 6 | >0·5 | 1·7805 | 0·5813 | 3·430 | 56·16 |
| 5 | 0·4–0·5 | 2·2617 | 0·5672 | 3·413 | 44·21 |
| 2 | 0·09–0·2 | 2·2305 | 0·7183 | 2·478 | 44·83 |

ceramic powder, there is such a relation, which can be presented as shown in Fig. 2, but with certain exceptions.

Fraction 1, which contained the smallest particles (up to 0·09 mm), showed the greatest resistance, related to the the pressure influence (the highest $\sigma_d$-value in Table 2), which could be a reflection of plastic deformation of its particles. Increase of particle diameters (from 0·09 mm to 0·5 mm and more) caused a decrease of powder resistance, as suggested by decreasing deformation stress values in the last column of Table 2. This might be a consequence of the increasing contribution of brittle deformation of particles. However, certain irregularity, concerning the order of fractions, can be noticed, i.e. fraction 2 (consisting of particles between 0·09 and 0·2 mm), has a very low value of deformation stress; lower than the value which might be expected according to the $d(\sigma_d)$-relation.

The reasons for the behaviour of fraction 2 should not be searched for in the chemical composition. From Table 1 it is obvious that the fractions do not differ significantly in their chemical compositions. Therefore, the same conclusion can be expected (and was proved) in the case of the phase composition. The differences among compressibilities of fractions are probably caused by some physical factors, i.e. by the 'geometry' at a particle level. It was proved (by SEM micrographs of initial powders, as well as by typical values of specific surface areas, given in Table 1) that particles of all sizes had rather irregular shapes and porosity. So, many vacancies and closed pores, inside each of the particles, could be expected. This is especially evident for fraction 2 and very well illustrated by the small weight of its particles, i.e. by the minimal value of its theoretical density (see Table 1).

## 5 CONCLUSIONS

The compaction process, for the fractions of investigated ceramic powder, can be described by the Heckel exponential equation. Consequently, this equation can be used for estimation of average deformation stress for particular fractions. A correlation between average diameter of particles, present in one fraction, and its deformation stress was found. It was shown that the increase of particle size causes a decrease of deformation stress and therefore improves compressibility of the fraction which contains such particles. However, some exceptions were noticed, i.e. particles between 0·09 mm and 0·2 mm have an average deformation stress smaller than the larger particles. It was supposed that many vacancies and closed pores inside these particles make them easy to crush, which significantly improves the compaction. So, there are other factors (besides particle diameter) that can influence the compaction process.

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