Influence of compacting times and pressures on rheological properties of alumina and quartz ceramic powder mixtures

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Abstract. In this research the compacting and rheological properties of alumina and quartz powder mixtures were examined depending on forming pressures and times. On the basis of the achieved relative compaction curves as function of forming pressure and time the authors could successfully describe the changes of rheological behavior and rheological model of these powder mixtures. With increasing the compression pressure of the alumina and quartz powder mixtures considerable changes of rheological properties could be observed.

In this work the authors have shown how the compacting deformations of cylindrical specimens are depending on compaction pressures and times. On the basis of their research the authors could successfully illustrate also how the rheological behaviors and models of alumina-quartz ceramic powder mixtures are changing in the compacting die cavities at low values of forming pressures.

1. Introduction

In our days the ceramic materials and ceramic matrix composites [1-25] are used in very wide range of industry and human life thanking to their excellent physical, mechanical, chemical and biological properties. Because of the variety of their chemical, physical and material compositions, several techniques and technologies are applied to develop ceramics and ceramic based composites, including sol-gel technology, spark plasma sintering, spray drying, injection molding, hot pressing and many other technological processes [26-49]. In spite of the wide variety of the production technologies generally the ceramic materials and ceramic based composites have hard crystal grains with high values of elastic modulus depending on their material compositions and structures [17, 50 and 51].

Today application of rheology is playing very important role both in materials science [52-72] and in technology processes [73-81] especially in forming technology. To prepare products from alumina and quartz ceramic powder mixtures there are several forming methods and technologies can be used including slip casting [82], injection molding [83] and pressing [45, 48, 84-87].

2. Materials and experiments

For the experiment were used alumina powder Nabalox 315 and silica powder from Fehérvárcsurgó. Before compacting the specimens the alumina powders were mixed with quartz powders and milled together in a laboratory planetary ball mill Retch PM 400 trough 20 minutes of each at 200 rotation/minute. The microstructure and size distribution of the used powders are shown in Figure 1.
and Figure 2. The so prepared powder mixtures were compacted through one-sided pressing to rods in a cylindrical die cavity with 10mm diameter and filling depth of 30mm on uniaxial hydraulic press.

![Surface micrograph and particle size distribution curve of the alumina](image1)

**Figure 1.** Surface micrograph and particle size distribution curve of the alumina

![Surface micrograph and particle size distribution curve of the quartz](image2)

**Figure 2.** Surface micrograph and particle size distribution curve of the quartz

During the experiments the compactions (displacements of pressing punch) were recorded by a computer as a function of compression pressures. In first stage the compaction deformations were studied at low compacting pressures and further the so pre-compacted powders in the die cavity were final compacted at 30bars, 60bars, 90bars, 120bars and 150bars in the hydraulic cylinder at final pressure values as shown in Table 1.

| Table 1. Final pressure values of compacting |
|---------------------------------------------|
| Pressure in the hydraulic cylinder | Pressure forces | Compacting pressures |
| bar  | N   | MPa  |
| 30   | 5966 | 76   |
| 60   | 11932| 152  |
| 90   | 17898| 228  |
| 120  | 23864| 304  |
| 150  | 29830| 380  |
3. Results and discussions
In first the compactions were studied at low compacting pressures and a typical such curve is shown in Figure 3. The compression chart shows the deformation and rheological behavior of the mixed alumina quartz powders under small values of forming pressures. In the first stage under a low compaction pressure the deformation is growing very quickly and can be regarded as almost ideally viscous material and characterized by Newton’s model. In the second stage the evolving stress causes the grains to roll on each other consequently filling the particle gaps, there by deformation occurs and the material can be modeled as row-linked viscous-elastic body. In the third stage the particles are continuing to fill the voids, meanwhile the compaction pressure is growing more quickly. In this stage the compaction is non-linear and can be modeled as parallel-linked viscous-elastic body, due to the frictions between the particles during their movement in the die cavity.

![Figure 3. The compression chart with the determined rheological behavior](image)

Finally in the fourth stage the particles of the ceramic powder are getting closer and closer to each other causing a significant increase in pressure meanwhile the compaction is relatively small. In this stage the ceramic powder mix behaves as a “quasi-plastic” material and can be modeled as it is shown above in Figure 3.

After the study the compression properties of alumina-quartz mix powders at low pressure the pre-compacted materials were final compacted without moving out from the die cavity at compacting pressures from 76MPa to 380MPa as it is shown in Table 1. The compaction deformation at different final compaction pressures are shown in Figure 4. At the first test when the final compression pressure was 76MPa the measurement has started before the pressing punch would have achieved in the die cavity the surface of the pre-compactied powder. Because of this the measured “compaction deformation” was higher than in reality.
Figure 4. The compaction deformations of cylindrical specimens are depended on forming pressure

It is obvious in the Figure 4 that increasing the compaction pressures from 152MPa up to 304MPa the compaction deformation of pre-compacted rod specimens have increased from 8mm up to 11mm. The further increase of compression pressure up to 380MPa didn’t influence on compaction deformation. These curves show that it is impossible to develop the density of the used ceramic powder mixtures in the cylindrical die cavity through further increment of compaction pressure.

In the next experiments (Figure 5) the pre-compacted ceramic rods were further compacted without moving out from the die cavity at compacting pressures from 76MPa to 380MPa, but the pressing punch was kept at the final compression pressure by 10-15sec. It is very well seen in Figure 5 that at the achieved compaction deformations the compacting pressures have decreased with increasing the time thanking to the mechanical stress relaxation inside of the powder mixtures in the die cavity. This phenomena informs us also that the particles of the powder mix have continued their movements and orientations in the die cavity without changing their total geometrical volume.

Figure 5. The relationship between the forming pressure and time

4. Conclusions
From the realized by authors experiments it is obvious that at low compaction pressures in the cylindrical die cavity the ceramic mixtures can be characterized with different rheological models depending on forming pressure.

The achieved results of the realized by authors examination show that it is impossible to develop the density of the used ceramic powder mixtures in the cylindrical die cavity through further increment of compacting pressure higher than 304MPa.
During uniaxial compaction of ceramic powders the mechanical stress relaxation is starting already inside of the die cavity.

There are further experiments are required to find the rheological model of ceramic powder mixtures in the cylindrical die cavities at final (high) compaction pressures.

Acknowledgement
The authors would like to acknowledge to IGREX Engineering Service Ltd for their support and help during this research work.

The described article was carried out as part of the EFOP-3.6.1-16-2016-00011 “Younger and Renewing University – Innovative Knowledge City – institutional development of the University of Miskolc aiming at intelligent specialization” project implemented in the framework of the Szechenyi 2020 program. The realization of this project is supported by the European Union, co-financed by the European Social Fund.

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