

UNIVERSITY OF MISKOLC FACULTY OF EARTH SCIENCE AND ENGINEERING MIKOVINY SÁMUEL DOCTORAL SCHOOL OF EARTH SCIENCES



# THE CONTROL OF DISPERSITY PROPERTIES AND PARTICLE DESIGN OF FINE-GRADED REFRACTORY RAW MATERIALS BY COMMINUTION OPERATIONS

Theses booklet of doctoral (PhD) dissertation

### László Tamás

Process Engineer

## Scientific supervisors

Dr. Ádám Rácz Prof. Dr. habil. Gábor Mucsi

MIKOVINY SÁMUEL DOCTORAL SCHOOL OF EARTH SCIENCES Head of the doctoral school: Prof. Dr. Péter Szűcs Professor, Doctor of the Hungarian Academy of Sciences

# I. The aim of the dissertation, scientific preliminaries

Our fundamental intention during development and improvement is to achieve these aims as efficiently as possible, where efficiency can be defined in various ways depending on the given circumstances. For the manufacturing industry, during the preparation of raw materials it is of critical importance that they reach the desired state with the lowest possible energy consumption. This is particularly true in the case of the Energy Intensive Industries (EIIs). However, in many cases, the raw materials produced with diverse technological processes and high energy intensity require additional mechanical process engineering preparation to obtain the product properties required by the area of use. Given that the most basic and at the same time the most energy-intensive operation of mechanical process engineering is comminution, the investigation of the basic phenomena occurring during particle size reduction processes is of paramount importance. Nearly 2% of the world's total electrical energy consumption is spent on various preparation processes, according to Napier-Munn (2015), of which 80% is taken up by comminution processes (Abouzied and Fuerstenau, 2009). Tromans and Meech (2002) also came to a similar conclusion, and also found that although 65-80% of the energy used during mining is spent on crushing, only 1-2% of the energy consumption can be attributed to the newly produced and increased free surface area of the material. Under the current circumstances, according to Kawatra and Eisele (2005), there is a great opportunity to develop and/or replace energy-intensive and currently energyinefficient particle size reduction processes.

The focus of my scientific dissertation is on the particle design of artificially produced refractory raw materials with high alumina content and special mechanical properties by comminution operations. The comminution of these materials is an extremely difficult task due to their hardness (corundum: 9 Mohs, mullite: 6-7) and abrasive properties. Corundum has the largest production volume in this group of materials, the above-mentioned properties of which allow it to be used in very diverse areas of application in various industries. Its most important industrial application is in the production of refractory materials due to its high melting point, in the chemical industry due to its chemical resistance, and its extraordinary abrasive properties are utilized in the field of sand blasting and polishing. It is also important to mention high-volume industries such as the aerospace and aircraft industry, medical technology and the laminate industry to produce various wear-resistant surfaces, which use corundum fractions that meet particularly strict criteria in their production processes. Mullite is mainly used in the ceramic industry for the production of refractory glazes and enamels.

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During my research, I sought an answer to the question of how artificially produced refractory raw materials with a high alumina content and the properties expected by the user industry can be characterized by specific property functions and their properties can be controlled by using innovative grinding technologies instead of conventional grinding processes.

The breakage of granular materials occurs as a result of several, currently poorly understood microprocesses. The main reason for this is that these phenomena are influenced by several parameters at the same time, and due to their combined effect, they will have a complex effect on the properties of the resulting product particles. In the field of comminution by compression, three main cases are distinguished, which were investigated extremely thoroughly by Prof. Klaus Schönert, breaking down to the level of basic phenomena. He presented single-particle breakage in his doctoral dissertation written in 1966, in which he discussed in detail the breakage of particles of different materials and their energy requirements, the dependence of single-particle breakage on particle size, the probability of single-particle breakage at different energy levels, and the size distribution and specific surface area of product particles, thereby laying the foundations for the scientific description of single-particle breakage. In his dissertation, he recognized that material properties influence the fracture and fracture strength of particles, as well as their tendency to deform. He reported that fracture strength can be defined by the energy required to fracture a particle, as well as its maximum tensile strength. Schönert's 1966 doctoral dissertation made it clear that a complete understanding of fracture, and in particular single-particle breakage, requires the investigation of the most elementary fracture processes. In order to understand the complex effect of the large number of particle-particle interactions that occur during confined-particle-bed breakage on the comminution process, Schönert recognized that proper in-depth investigation of the most fundamental microprocesses and phenomena is crucial. In 1977, Professor Klaus Schönert and his team at the University of Karlsruhe began to investigate confined-particle bed comminution in a piston press. The piston press apparatus and methodology used by Schönert are still the standard procedure for investigating stress by compression of a particle bed. His scientific results were embodied in his 1977 German patent and his 1982 American patent. He found that slow compressive stress causes the particle bed to undergo a volume reduction, which allows for favorable energy-efficient particle size reduction. In the case of open-particle-bed breakage, the particles subjected to compression may be displaced outside the compression zone under the load, thus reducing the efficiency of the crushing, which is illustrated in Figure 1. Schönert (1979) finally concluded that the most efficient compression-based fracture mechanism from an energetic point of view is single-particle breakage, followed by confined and finally open particle-bed breakage.

In 1984, Schönert utilized his 1977 and 1982 patents and built a laboratory-scale HPGR (High Pressure Grinding Rolls) device with a cylinder diameter of 200 mm and a peripheral speed of 0.1-3 m/s with his research partner, with which he implemented continuous, high-pressure confined-particle-bed grinding (Schwechten and Schönert, 1984). The first industrial application of the new technology, due to its energy-sensitive nature, took place in the cement industry in 1985 (Schönert 1988). The efficiency of HPGR devices in the cement industry has been confirmed by several authors:

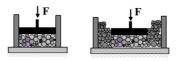


Figure 1. Confined and open particle-bed compression Source: Tomas (2014)

According to Wüstner (1986), 30% of the energy can be saved with a closed-loop HPGR ball mill system compared to single-stage ball mill process for cement grinding. Patzelt (1992) came to the same conclusion regarding the HPGR ball mill cycle for cement clinker grinding. Ellerbrock (1994) found that clinker comminution with an HPGR unit not only resulted in 15% energy savings, but also a 20% increase in capacity. It is important to note that while the energy efficiency of ball mills is strongly limited by the feed and target particle size, the specific grinding energy requirement can be reduced from 30-42 kWh/t (Marchal, 1997) to 20-30 kWh/t with an HPGR ball mill grinding system (Alsop and Post, 1995, Cembureau, 1997, Seebach et al., 1996). Madlool et al. (2011) found that energy efficiency improvements of up to 10-50% can be achieved when examining different HPGR - ball mill configurations. Good evidence of its applicability in the cement industry is that, according to a study by Daniel (2007), approximately 400 large-capacity HPGR units were operating at that time in the field of clinker grinding worldwide. Despite all this, the primary industrial applications of HPGR equipment were mainly in pre-grinding, but later the technology gained ground in hybrid comminution and industrial tasks requiring higher grinding fineness (Kellerwessel, 1993, 1996). The reliability, simplicity and high capacity of the equipment ultimately proved to be advantageous features that the mining industry had been seeking for a long time (Kodali et al., 2011).

As a result of extensive basic research and equipment developments, although the processes taking place during the application of the crushing mechanism and technology had not been clarified in all details, the first truly successful ore crushing application was carried out in the

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Los Colorados mine in Chile in 1998. At that time, only 14 pairs of high-pressure grinding rollers were operating in various mines around the world to increase the specific surface area of the ground material, and three for coarse crushing of various ores (Van der Meer, 2012). In 2013, according to Sesemann (2013), 130 HPGR units were operating in numerous ore and mineral preparation plants around the world.

HPGR equipment and the continuous high-pressure compression particle-bed comminution method created in them are based on the work of Professor Klaus Schönert and thanks to him, this approach spread in various industries requiring particle size reducing technologies, whose scientific background and description was based on his systematic piston press tests. As a result of these tests, he determined the minimum pressure value for effective particle-bed breakage at a particle-bed pressure of 50 MPa, which is ideally be 100-300 MPa depending on the material (Schönert, 1988). He introduced the concept of "interparticle breakage", i.e. the breakage between the particles that occurs during particle-bed comminution. During his scientific work, he established that the efficiency of breakage is most closely related to the energy absorbed from the equipment by the particle bed formed by the particulate material, which is the most relevant factor in the energetic aspect of the confined particle breakage (Schönert, 1991). A few years later, Schönert (1996) defined the criteria system of the ideal particle bed, which he bound to the following conditions based on his research: (1) The existence of a stochastically homogeneous particle bed; (2); A homogeneously compacted particle bed; (3) The volume or mass of the particles being stressed is known; (4) A negligible wall effect from the point of view of the breakage process. The researcher found that these conditions can be achieved by parallel cylindrical pressing surfaces in a piston press equipment, in the case where the particle-bed thickness is at least six times the maximum feed particle size and the bed diameter is at least three times its height.

Schönert (1996) found that, for given specific absorbed energy levels, the fraction of broken particles increases as the number of particle stacks is decreased, i.e. the probability of broken particles rises, as shown in Figure 2. Similar results were obtained by Gutsche and Fuerstenau (1999) and Pourghahramani and Forssberg (2007). Schönert (1996) saw the following reasons for this phenomenon: (1) increasing the particle-bed thickness results in increased friction loss due to the increase in the number of particles and thus the number of contact points, (2) the number of elastic and plastic deformations also results in a process with a higher total energy requirement due to the increased contact points, and (3) the formation of micro-cracks in the particles also requires additional energy. It is important to mention that the increase in the number of contact points has a positive effect on the formation of a cubic particle shape and a low particle-width-to-particle-length ratio of the fraction. According to Briggs and Evertsson (1998), it is therefore advisable to use choke feeding in cone crushers

so that a particle bed is created during the comminution process, which is favorable to the formation of a blocky, cubic particle shape in the product. Schönert (1996) also showed that although the friction between the walls of the piston press and the particles significantly influences the pressure distribution in the particle bed, the effect of the pressure distribution on the comminution is not significant.

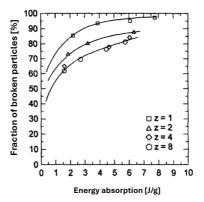


Figure 2. 3.2–4 mm effect of absorbed energy on the broken fraction of quartz particles Source: Schönert (1996)

It can be seen that many researchers have reached essentially identical results regarding the effect of particle-bed thickness in their work, from which one of the most important conclusions that can be drawn is that the energetic efficiency of comminution and the particle-bed thickness are inversely proportional to each other, as shown in Figure 2.

In addition to the particle-bed thickness exposed to stress, the next important basic parameter is the particle-bed pressure, which stresses the particles located in the bed. The particle-bed pressure has a direct effect on pressure distribution within and between the particles, and thus on the fracture mechanism they will undergo. Kanda et al. (1990) found that during the particle- bed grinding of narrowly classified quartz particles with a particle size of 1.19–1.41 mm, a higher applied particle-bed pressure will result in a higher probability of fracture and reduce the median particle size of the resulting product. Gutsche and Fuerstenau (1999) observed during their measurements that by increasing the particle-bed pressure, the quartzite particles with a particle size of 1.68–2.38 mm in the bed at first compacted, trying to achieve the highest possible bulk density and the associated space filling under given pressure

conditions. Under low pressure conditions, small fragments break off from the feed particles, which fill the voids in the particle bed, and then the bed compacted, which is illustrated in Figure 3. It can also be observed that from the pressure range of 50-100 MPa, where the tendency of the bed compaction becomes much lower, the process of effective crushing and particle size reduction begins, which is accompanied by a slower rate of particle-bed compaction. Mütze (2016) reported on fine limestone, quartz and silicon carbide samples with particle sizes of 10 and 50 µm, showing that the elastic energy stored in the particles increases with increasing bed pressure, which leads to a loss of fracture energy, thus reducing the energy efficiency of the comminution process.

In conclusion, despite the minimum particle-bed pressure of 50 MPa according to Schönert (1988), it should be noted that using excessively high particle-bed pressure can lead to reduced energy and breakage efficiency. It is therefore extremely important that the optimal particle-bed pressure is used, knowing the given comminution goals, in order to avoid energy waste and increased equipment wear.

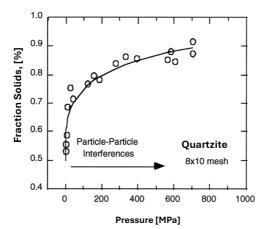


Figure 3. Effect of pressure on particle-bed compaction Source: Gutsche. and Fuerstenau (1999)

In addition to the particle-bed thickness and pressure, the stress loading rate is also a fundamentally important breakage parameter, where, according to Kerber (1984), a lower stress rate favors the formation of a more compact particle bed, while a higher stress rate leads to increased friction losses at the contact points of the particles. In the piston press experiments of Mütze and Husemann (2008), the stress rates of 0.05 cm/s and 10 cm/s showed

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significantly different energy absorption values at different pressure values, which is illustrated in Figure 4. The higher stress loading rate is always associated with better energy absorption in the pressure interval of 175–400 MPa, which is a key measure of the grinding efficiency. However, it is also worth considering that a loading rate of 200 times was required to reach this.

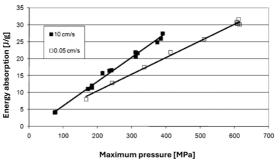


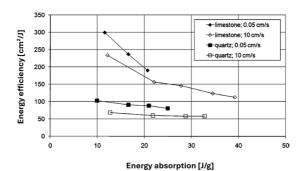
Figure 4. Effects of stress loading rates on energy absorption at different particle-bed pressures Source: Mütze and Husemann (2008)

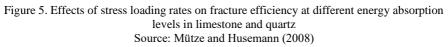
The energy efficiency of the breakage shows a decreasing trend with increasing compression velocity, which is illustrated in Figure 5. Both the less hard limestone and the much harder 10-15  $\mu$ m quartz particle fractions can be fractured more efficiently using lower compression velocities, because the surface area increase per unit of absorbed energy is greater.

The investigation of confined particle-bed breakage using piston press experiments has attracted the attention of many authors over the past four decades, resulting in an extensive literature in this field of science. The presented series of experiments and the resulting scientific publications have examined this comminution method in various aspects using phenomenological methods. Although many authors have investigated quartz, which has hard and brittle fracture and abrasive properties, as a model material in their research, only a few researchers (Cooper and Eaton, 1962, Reichardt and Schönert, 2003, Karimi and Djokoto, 2012, Pedrosa et al., 2019) have used materials with extraordinary mechanical properties such as corundum, silicon carbide and zirconium silicate as model materials in their bed comminution research.

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It is also important to mention that among the investigations of the dispersity properties of the product particles created by confined particle-bed breakage, particle shape and the resulting bulk density have not been the focus of researchers in the past four decades, and according to Pedrosa et al. (2019), HPGR equipment is not used for corundum grinding anywhere in the world.

## Based on the literature the following conclusions can be drawn:

- There are few scientific publications dealing with the confined particle-bed comminution by slow compression of extremely high-mechanically strong and particularly abrasive materials such as corundum (Cooper and Eaton, 1962, Reichardt and Schönert, 2003, Karimi and Djokoto, 2012, Pedrosa et al., 2019). Pedrosa et al. (2019) note that they are not aware of any HPGR crushing equipment being used anywhere in the world for corundum crushing in 2019. Overall, the authors of all four publications have concluded that the confined bed comminution method provides a suitable technological solution for the crushing of high-hardness, extremely resistant and abrasive materials. Despite this, there are few scientific publications in this area, and the industrial application of HPGRs has only spread to materials with less abrasivity and lower mechanical strength.

- The scientific literature mainly discusses the modification of the dispersity and application properties of granular materials by confined particle-bed comminution by examining the particle size distribution and specific surface area. The effect of the particle shape of the feed material on fracture has been investigated in many cases,; however, only a small number of publications examine the shape of the product particles, while in none of the cases were the test materials used as raw materials for the refractory industry.

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- The effect of impact on a solid surface and particle-particle collision on particle shape has been investigated by many researchers; however, in none of the cases have they dealt with a special refractory raw material such as mullite.

- The scientific literature does not examine the effects of a bed thickness that is thinner in depth than the so-called ideal particle bed, nor does it mention what dispersity property changes can be achieved by reducing the particle-bed thickness, among which I would highlight the particle shape and the resulting bulk density.

# II. Objectives of the research work

- 1. Process control of the particle shape and particle fraction's bulk density of high-aluminacontent molten products of different particle sizes and its scientific foundation, with special regard to particle-bed comminution by slow compression.
- 2. Investigation of the effect of the breakage stress environment on the dispersity properties of high alumina content molten products, to establish a basis for conscious product design. (Confined-particle-bed breakage of corundum and impact-stress related breakage in the case of mullite.)
- 3. Determination of the relationships between the property functions and dispersity properties required by the application areas and the process parameters influencing the confined-particle-bed comminution method.
- 4. The main aim is to design and develop a high-pressure grinding roller pair for research purposes, which can be used to produce the desired target product in the right quality and quantity, by confined-particle-bed comminution. As a result of the equipment development, my goal was to create a high-pressure grinding roller pair that can effectively grind corundum using a transient particle breakage method that simultaneously ensures effective particle size reduction resulting from single particle breakage or low particle-bed thickness and has the favorable particle dispersity properties associated with the particle-bed comminution method by slow compression.

# **III. Experimental**

The research, design and development work I have carried out includes the study of Hungarian and international literature, the development of experimental and test methods included in the dissertation, laboratory and plant experimental tests, collection of data, the evaluation of the obtained results, and the drawing of conclusions.

The scientific results included in my dissertation are based on systematic laboratory and plant experiments, which I carried out at the Institute of Raw Material Preparation and

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Environmental Technology of the University of Miskolc and at the Újrónafő site of Refra-System Kft.

During the research, I have summarized the systematically carried out tests and their results in three main chapters. These are: (1) Piston press experimental results; (2) Development and measurement results of a semi-industrial HPGR device; (3) Results of horizontal and vertical shaft impact crushers.

### Materials and methods

In my first experiment, I carried out my research work with a piston press using a systematically constructed experimental and measuring system, during which I examined the effects of altering the particle-bed thickness (h) and the piston pressure (p) on the dispersity properties of the products in corundum. For the measurements, I consciously selected and prepared the feeding model material into a 1-3 mm corundum fraction, producing it by single-particle breakage and a linear sieving machine, the 10, 50 and 90 % particle size of which was 1.195 mm, 1.76 mm and 2.69 mm. During my experiments, I used particle-bed thicknesses of h=5, 10, 15, 20 and 25 mm and piston pressures of p=125, 175, 225 and 250 MPa while applying a pressing speed of 31.5 mm/s.

I used a Camsizer X2 device, which uses a dynamic image analysis method, to determine the particle size and shape distribution of the particles by dry dispersion. According to my preliminary results, the particle sphericity (SPHT) correlates more strongly with bulk density than with the particle length-width (b/l) ratio of the given narrowly classified fraction. Accordingly, I used the SPHT parameter to describe the particle shape, which is calculated by the device as follows:

$$SPHT = \frac{4\pi \times A}{P^2}$$

where SPHT is sphericity of the tested sample, P is the perimeter of the particle and A is the projected area of the particle. A perfect sphere has a sphericity of 1, and all other particle shapes will result in SPHT<1.

I introduced novel dynamic metrics to describe the particle shape and bulk density changes of fractionated products produced by particle-bed comminution with different breakage parameters. SPHT<sub>increase</sub> and  $\rho_{bulk,increase}$  express the increase in particle shape and bulk density changes, respectively, compared to single-particle breakage for a given product fraction produced by confined-particle-bed comminution. The  $\eta$  value is an efficiency number that characterizes the comminution and particle shape formation, which provides information about the mass yield of <1 mm product fractions and their cumulative sphericity increase. This  $\eta$  value shows the importance of the fact that it is not enough that a product contains

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particles with appropriate particle shape and bulk density under given production parameters, it is at least as important that the proportion of these particles in the product is also high. I have shown the dependence of the particle size distribution, sphericity and bulk density of the broken fractions (<0.15, 0.15-0.3, 0.3-0.5, 0.5-1 mm) on the applied piston pressure and particle-bed thickness through figures, using the above-presented metrics and the well-known basic process engineering functions.

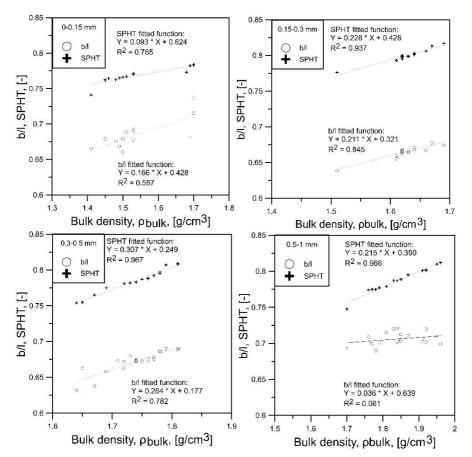
Subsequently, using my experiences and the measurement results of my experiments performed with the piston press, I developed a semi-industrial-sized, high-pressure grinding roller pair suitable for conducting scientific studies, with which continuous comminution of <5 mm corundum particles in a confined particle bed can be performed. The basis of the equipment was a high-pressure roller compactor. Through experimental studies, I have clarified the technical and scientific relationships that exist between the technological parameters of continuous, high-pressure, confined-particle-bed comminution (s - optimal gap = 5 mm, P<sub>spec</sub> – specific cylinder pressure = 6.88; 12.19; 15.63 and 20.31 MPa and V<sub>circ</sub> – cylinder circumferential velocity = 72, 108 and 143 mm/s, obtained from previous studies) and the yield of the target product and its fractions, as well as the change in particle shape and bulk density. To illustrate this, I have introduced the variable  $\Omega$ , which is an efficiency measure characterizing breakage and particle shape formation.

Finally, I examined the effect of two basic comminution operating parameters ( $V_{circ}$  - rotor circumferential speed = 37.7, 47.1, and 56.5 m/s and m - feed mass flow = 3.25, 4.76, and 6.86 t/h) on selected and prepared 1–3 mm mullite particles in VSI (Vertical Shaft Impactor) and HSI (Horizontal Shaft Impactor) equipment. The model material prepared as feed was prepared by single particle breakage and a linear sieving machine, with 10, 50, and 90% particle sizes of 1.14 mm, 1.78 mm, and 2.79 mm respectively.

The dependence of the particle size distribution, sphericity and bulk density of the broken fractions (<0.15, 0.15-0.3, 0.3-0.5, 0.5-1 mm) on the applied operating parameters was shown in figures. The laboratory sieve analyses were performed with a Retsch AS 200 TAP device, during which the sieves were shaken at 280 rpm and subjected to 150 impacts/min. The standardized procedure according to MSZ 6506-84 was used to determine the bulk density of the different corundum and mullite fractions. The particle shape alteration was illustrated with images taken with a Zeiss Axio Imager M2M device.

# **IV. Scientific results, theses**

1. From the analysis data obtained by dynamic image analysis method, I determined the functional relationship between the average particle shape descriptive parameters (SPHT, b/l) and the bulk density of the <0.15; 0.15-0.3; 0.3-0.5 and 0.5-1 mm corundum fractions. I proved that the particle sphericity (SPHT) is a suitable particle shape descriptive parameter to describe the relationship between particle shape and bulk



density in the case of the above-mentioned corundum fractions, which is supported by the test results in Figure 6.

Figure 6. Relationship between the shape descriptive parameters b/l nd SPHT and the bulk density

### 2. I developed a novel experimental and evaluation method to investigate and describe the particle shaping process occurring during confined-particle-bed breakage by slow compression.

I have introduced the following novel metrics to describe the effectiveness of particle shaping:

$$SPHT_{\rm inc} = \frac{SPHT_1 - SPHT_0}{SPHT_0} \times 100$$

where  $SPHT_{inc}$  shows the increase in sphericity of a given fraction,  $SPHT_0$  is the sphericity of the fraction produced by single-particle breakage, while  $SPHT_1$  is the sphericity of the fraction of the same nominal particle size produced by confined-particle-bed breakage.

$$\eta = \frac{(\Sigma SPHT_{\rm inc} \times K)}{100}$$

where  $\eta$  is a measure of the efficiency of breakage and particle shaping,  $\Sigma SPHT_{inc}$  is the sum of the sphericity of the particle fractions, and K is the mass yield of the <1 mm product fraction.

$$\Omega = \frac{(SPHT_{inc} \times k)}{100}$$

where  $\Omega$  is a measure of the efficiency of breakage and particle shaping for a given product fraction, SPHT<sub>inc</sub> is a measure of the sphericity of the given product fraction, and k is the mass yield of given fractions in the <1 mm product.

3. I have proven with static model tests that confined-particle-bed breakage by slow compression can be utilized for particle shaping. The change in particle shape leads to an increase in the bulk density of the product fractions; therefore, several product properties of <1 mm corundum grains can be controlled simultaneously (particle size, particle shape, bulk density).

4. I have proven with static model tests that by varying the applied piston pressure and particle-bed thickness, efficient shaping of narrowly classified corundum products with different size fractions can be achieved during the comminution process.

a. In the case of <0.3 mm corundum products, the greatest increase in sphericity and bulk density can be achieved by using 5-10 mm and 225-250 MPa, while in the production of 0.3-0.5 mm products, 20 mm and 175-250 MPa, and in the production of 0.5-1 mm products, 20 mm and 125-175 MPa particle-bed thickness and particle-bed pressure.

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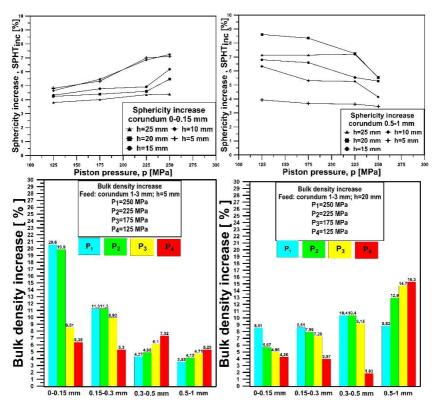


Figure 7. The effect of the applied particle - bed thickness and piston pressure on the sphericity increase of the 0-0.15 and 0.5-1 mm fractions and the effect of piston pressure on the bulk density increase.

b. Furthermore, with the  $\eta$  metric shown in Figure 8, I demonstrated that the transient breakage method, which is available between the bed thickness of single-particle and ideal particle-bed breakage (5-15 mm), produces a larger quantity and more spherical <1 mm product in all cases by increasing the particle-bed pressure.

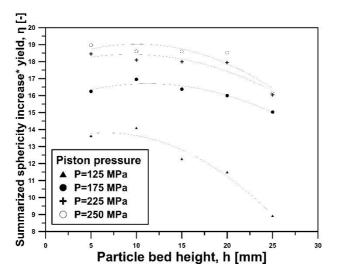


Figure 8. Effect of piston pressure and particle-bed thickness on the \eta parameter

5. Based on the results of static model tests, I developed a continuously operating HPGR device in which the breakage and simultaneous shaping of 1-3 mm corundum particles can be consciously controlled.

6. I determined the process engineering and scientific relationships of the semiindustrial HPGR device that exist between the technological parameters of continuous confined particle bed comminution (optimal gap size - s,  $P_{spec}$  – specific cylinder pressure and  $V_{cir}$  – cylinder peripheral speed obtained from previous studies) and the yield of the target product and its fractions, as well as the change in particle shape and bulk density.

- a. I found that the application of V<sub>ker</sub>=72 mm/s resulted in continuously increasing  $\eta$  values (Figure 9 )with increasing specific cylinder pressure in the interval P<sub>spec</sub>=6.88–20.31 MPa,; therefore, the application of the lowest tested loading rate is the most appropriate for particle shaping performed simultaneously with comminution of the product particles.
- b. Using the  $\Omega$  quality indicator (Figure 10), I determined the optimal production conditions for the <0.15; 0.15–0.3; 0.3-0.5 and 0.5–1 mm corundum product fractions in the self-developed HPGR equipment, in the examined intervals.

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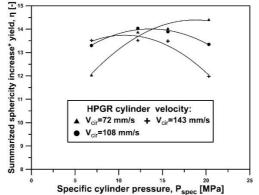


Figure 9. Effect of specific cylinder pressure and peripheral velocity on the parameter \eta

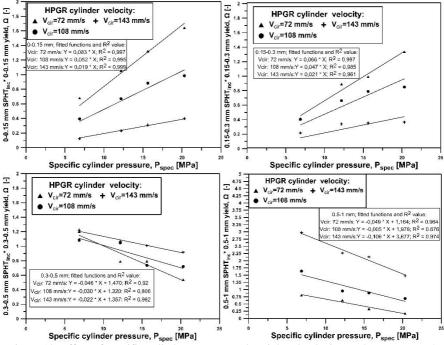


Figure 10. Effect of specific cylinder pressure and peripheral velocity on the parameter  $\Omega$ 

c. I have also found that the following settings are suitable for increasing the bulk density of <0.15; 0.15–0.3; 0.3–0.5 and 0.5–1 mm corundum products in the HPGR equipment, which is shown in Figure 11.

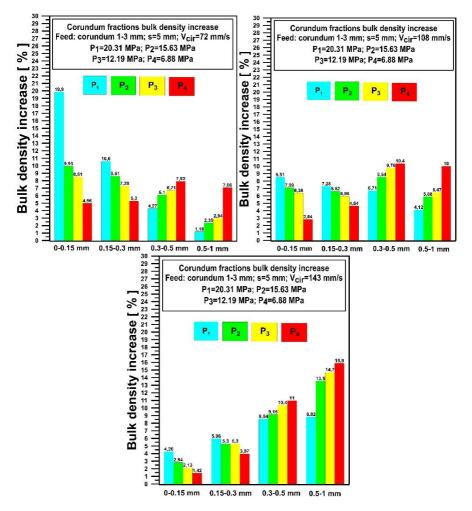


Figure 11. Effect of specific cylinder pressure on the bulk density increase of product fractions when using cylinder peripheral speeds of 72, 108 and 143 mm/s

7. I developed a calculation method based on breakage probability as a result of my confined-particle-bed compression experiments; with this, the relationship between the pressure applied during static particle-bed loading and the particle-bed pressure developed in the HPGR can be estimated.

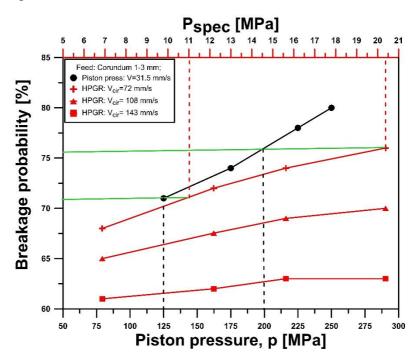


Figure 12. Conversion of HPGR specific cylinder pressure to piston press pressure values using the breakage probability

8. I have proven that the vertical shaft impact crusher (VSI) is a more suitable crusher for the comminution and simultaneous particle shaping of the 1-3 mm mullite fraction in the impact crusher than the horizontal shaft impact crusher (HSI) due to the higher product yield and cumulative sphericity increase, which can be explained by the greater number of particle-particle collisions occurring in the equipment.

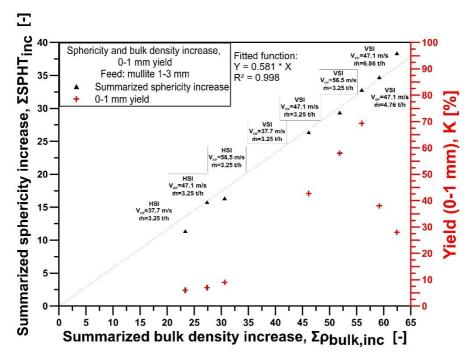


Figure 13. Relationship between the cumulative sphericity and bulk density increases with the yield of <1 mm product fractions

# VI. References

Abouzied A. M., Fuerstenau D. W. (2009): Grinding of mineral mixtures in high-pressure grinding rolls. International Journal of Mineral Processing, v. 93, p. 59-65. 10.1016/j.minpro.2009.05.008

Alsop. P.A. and Post. J.W. (1995): The Cement Plant Operations Handbook, Tradeship Publications Ltd., Dorking, UK

Briggs. C. and Evertsson. M. C. (1998): Shape Potential of Rock, Minerals Engineering, Vol. I1, No. 2, pp. 125-132

Cembureau. (1997): Best Available Techniques for the Cement Industry, Brussels

**Cooper.** A. R. and **Eaton**. L. E. (1962): Compaction Behavior of Several Ceramic Powders. Journal of the American Ceramic Society, 45(3), 97–101. doi:10.1111/j.1151-2916.1962.tb11092.x

University of Miskolc

Daniel. M. J. (2007): Energy efficient mineral liberation using HPGR technology. Doctoral dissertation, University of Queensland.

Ellerbrock. H.-G. (1994): Gutbett-Walzenmuhlen, Zement-Kalk-Gips 2, 75-82.

**Gutsche**. O. and **Fuerstenau**. D. W. (1999): Fracture kinetics of particle bed comminution ramifications for fines production and mill optimization, Powder Technology 105, 113–118.

Kanda. Y., Takahasi. S. and Sakaguti. N. (1990): A compressive crushing of powder bed- a consideration of size distribution of product and specific energy to produce fine particles. Powder Technology, 63 (1990), 221-227.

Karimi. H. R. and Djokoto. S. S. (2012): Instrumentation and modeling of high-pressure roller crusher for silicon carbide production. The International Journal of Advanced Manufacturing Technology, 62(9-12), 1107–1113. doi:10.1007/s00170-011-3871-8

Kawatra S.K. and Eisele T.C. (July 2005): "Optimization of Comminution Circuit Throughput and Product Size Distribution by Simulation and Control". Final Technical Report, Department of Chemical Engineering, Michigan Technological University, DOE Award Number: DEFC26-01NT41062

Kellerwessel. H. (1993): High pressure particle bed comminution of mineral raw materials. Aufbereitungs-Technik 34 (5), 243–249.

Kellerwessel. H. (February 1996): High pressure particle bed comminution, state of the art, application, recent developments. Minerals Engineering Journal, 45–52.

Kerber. A. (1984): Einfluß der Beanspruchungsgeschwindigkeit, Profilierung und Rauhigkeit auf die Einzelkorn-Druckzerkleinerung, dissertation, Universität Karlsruhe.

Kodali. P., Dhawan. N., Depci. T., Lin. C. and Miller. J. (2011): Particle damage and exposure analysis in HPGR crushing of selected copper ores for column leaching. Minerals Engineering, 24, 1478–1487. <u>https://doi.org/10.1016/j.mineng.2011.07.010</u>

**Madlool**. N.A., **Saidur**. R., **Hossain**. M.S. and **Rahim**. N.A. (2011): A critical review on energy use and savings in the cement industries, Renewable and Sustainble Energy Reviews 15, 2042–2060.

**Marchal**. G. (1997): Industrial Experience with Clinker Grinding in the Horomill Proc. 1997 IEEE/PCA Cement Industry Technical Conference, XXXIX Conference Record, Institute of Electrical and Electronics Engineers: New Jersey.

Mütze. T. and Husemann. K. (2008): Compressive stress: Effect of stress velocity on confined particle bed comminution, Chemical Engineering Research and Design, 8(6) (, 379–383

**Mütze**. T. (2016): Modelling the stress behaviour in particle bed comminution, International Journal of Mineral Processing, 156, , 14–23.

Napier-Munn T. J. (2015): Is progress in energy-efficient comminution doomed? Minerals Engineering, v. 73, p. 1-6.

Patzelt. N. (1992): High pressure grinding rolls, a survey of experience. IEEE Cement Industry Technical Conference, Dallas/Texas 10 (14),

**Pedrosa**. F. J. B., Bergerman M. G., Segura-Salazar, J. and Junior H. D. (2019): HPGR as alternative to fused alumina comminution route: an assessment of circuit simplification potential, REM - International Engineering Journal, 72(3), 543-551, <u>http://dx.doi.org/10.1590/0370-44672018720193</u>

**Pourghahramani**. P. and **Forssberg**. E. (2007): The characterization of structural changes in hematite ground in a confined particle bed using Rietveld analysis, International Journal of Mineral Processing 83, 47–59.

**Reichardt**. Y. and **Schönert**. K. (2003): Interpartical Breakage on Fine Hard Materials by Single and Multiple Compression. Chemical Engineering & Technology, 26(2), 191–197. doi:10.1002/ceat.200390028

Schönert. K. (1966): Einzekorn-Druckzerkleinerung and Zerkleinerungskinetik Untersuchungen an Kalkstern-. Quarz- and Zementklinkerkornern des GroBenbereiches 0,1-3 mm, Diss,TH Karlsruhe

Schönert. K. (1977): A method for fine and very fine comminution of brittle material behavior materials; Patent number: DE2708053A1

Schönert. K. (1979): Energetische aspekte des Zerkleinerns spröder Stoffe. Zement-Kalk-Gibs, v. 32. p. 1-9.

Schönert. K. (1982): Method of fine and very fine comminution of materials having brittle behavior. Patent number: US4357287A

Schönert. K. (1988): A First Survey of Grinding with High-Compression Roller Mills, International Journal of Mineral Processing, 22, 401-412.

Schönert. K. (1991): The characteristic of Comminution with High Pressure Roller Mills, KONA No.9, 149-158.

Schönert. K. (1996): The: influence of particle bed configurations and confinements on particle breakage, International Journal of Mineral Processing, 44-45 (1996) I – 16.

Schwechten. D. and Schönert. K. (1984): Gutbett-Zerkleinerung mit grossen Pressungen von mineralischen Rohstoffen. In: Fortschritte in Theorie und Praxis der Aufbereitungstechnik, Freiberg Preprints, pp. 116-120.

Sesemann. Y., Broeckmann. C. and Höfter, A. (2013) A new laboratory test for the estimation of wear in high pressure grinding rolls. Wear, 302(1-2), 1088–1097. doi:10.1016/j.wear.2012.10.022

Tomas. J. (2014): Folien\_MVT\_2neu Mechanische Verfahrenstechnik - Partikeltechnologie Zerkleinerung

**Tromans** D. and **Meech** J.A. (2002): A Fundamental Analysis of Fracture Mechanics of Minerals during Comminution. Research being conducted at The Centre for Environmental Research in Minerals, Metals and Materials (CERM3), UBC. Via the internet, January 2023. http://www.mining.ubc.ca/cerm3/energy%20efficiency.html

**Van der Meer**. F.P. and **Maphosa**. W. (2012): High pressure grinding moving ahead in copper, iron, and gold processing, The Journal of The Southern African Institute of Mining and Metallurgy, Volume 112, 1.

Von Seebach H. M., Neumann. E. and Lohnherr. L. (1996): State-of-the-art of energy-efficient grinding systems. Zement-Kalk-Gips 49(2):61–67.

Wüstner. H. (1986): Energy-saving with the roller press comminution process. World Cement, 94-96.

## VI. List of publications

#### Papers in international journals

 Tamás. L. and Rácz. Á. (2023): Effect of material bed compression on white fused corundum product particle shape and bulk density. Open Ceramics 16 Paper: 100489 8 p. https://doi.org/10.1016/j.oceram.2023.100489

#### Papers in international conference proceedings:

- Tamás. L. and Rácz. Á. (2023) : Effect of the impact breakage type on the properties of White Fused Mullite particles. In: PARTICLE TECHNOLOGY FOR SUSTAINABLE PRODUCTS -BOOK OF ABSTRACTS. (2023) p. 711, 26-28 Sept. Nuremberg, Germany.
- Tamás. L. and Rácz. Á. (2024): The effect of non-ideal particle bed breakage on the particle shape of white fused corundum. In: Faitli J. (ed.), Book of Extended Abstracts of the 18th European Symposium on Comminution & Classification: ESCC 2024. (2024) ISBN:9786156018250 pp. 94-97. 27-29 June 2022, Hungary, Miskolc.

#### Papers in Hungarian journals:

- Rácz. Á., Tamás. L. (2019): Development of a laboratory scale continuous dry stirred media mill. ÉPÍTŐANYAG: JOURNAL OF SILICATE BASED AND COMPOSITE MATERIALS 0013-970X 2064-4477 71 (3) pp. 92-96. <u>10.14382/epitoanyag-jsbcm.2019.17</u>
- Tamás. L. and Rácz. Á. (2021): Comparison of particle size and shape distribution of corundum produced by industrial ball mill and material bed compression. MULTIDISZCIPLINÁRIS TUDOMÁNYOK: A MISKOLCI EGYETEM KÖZLEMÉNYE 2062-9737 2786-1465 11 (5) pp. 59-67 2021. 10.35925/j.multi.2021.5.6
- Tamás. L. and Rácz. Á. (2022): Material bed compression experiments and the examination of the bulk density of the product. GEOSCIENCES AND ENGINEERING: A PUBLICATION OF

THE UNIVERSITY OF MISKOLC 2063-6997 10 (15) pp. 110-124 2022 10.33030/geosciences.2022.15.110

#### Papers in Hungarian conference proceedings:

 Tamás. L. and Rácz. Á. (2022): COMPARISON OF SINGLE PARTICLE BREAKAGE BY COMPRESSION LOADING AND A BREAKAGE BY MATERIAL BED COMPRESSION LOADING WITH THE EXAMINATION OF THE DISPERSITY PROPERTIES OF THE PRODUCTS, Molnár Dániel. Tavaszi Szél 2022 / Spring Wind 2022 Tanulmánykötet I. (2022) ISBN:9786156457134 pp. 220-234

#### Conference presentations:

- Tamás. L. and Rácz. Á. (2021): Anyagágy összenyomási kísérletek and a termékfrakciók halmazsűrűségének vizsgálatai. Doktoranduszok Fóruma 2021.11.18. Miskolc.
- Tamás. L. and Rácz. Á. (2021): Comparison of particle size and shape distribution of corundum produced by industrial ball mill and material bed compression. MicroScience-MicroCAD International Multidisciplinary Scientific Conference. Miskolc
- Tamás. L. and Rácz. Á. (2022): Comparison of single particle breakage by compression loading and a breakage by material bed compression loading with the examination of the dispersity properties of the products. Spring Wind Conference 06.05.2022. Pécs.
- 11. **Tamás**. L. and **Rácz**. Á. (2023): Effect of impact breakage type on the properties of white fused mullite particles. 26-28 Sept. 2023. Particle Technology, Nuremberg, Germany.
- Tamás. L. and Rácz. Á. (2024): The effect of non-ideal particle bed breakage on the particle shape of white fused corundum. 18th European Symposium on Comminution & Classification: ESCC 2024. 27-29 June 2022, Hungary, Miskolc.