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DEVELOPMENT OF TESTING METHODS FOR DETERMINATION OF FINE MATERIALS GRINDABILITY AND ENERGY DEMAND OF GRINDING COMPLETED UNDER SPECIAL CIRCUMSTANCES

Theses of PhD dissertation

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I. Introduction

Grinding is the fine of comminution, which produces < 0.5 mm particles, mostly < 100 µm, generally from larger than 5 mm feed size. This process has a very important role in the field of industry and agriculture, such as ore- and mineral mining, chemical-, pharmaceutical-, cement- and mineral processing industry, furthermore food industry, animal food production as well as treatment of biomass and different kinds of waste.

The comminution, grinding – especially fine grinding - is a very energy consuming process. According to literature 3…5 per cent of the total energy consumption of developed countries is directed to crushing and grinding [1,2]. Hence, the determination of specific grinding energy has a great practical value.

The grindability and resistance against mechanical stress of materials to be ground is of great importance from characterization point of view. Namely, this property affects significantly the behavior of mills, the efficiency of grinding process, the power requirement of grinding, etc… The dimensioning and specific energy demand's determination of mills are based on the knowledge of grindability of material.

Grindability is generally characterized by the grinding work of one unit weight or volume of material. It is determined in a proper apparatus under exactly defined conditions. For the dimensioning, the working capacity of the mill has to be known beside grindability.

However, several problems arise during mill dimensioning. Some of them are originated from the determination of grindability of the brittle material. Namely, different grindability indices – Bond-work index (W_i), Hardgrove Grindability Index (HGI), Zeisel specific grindability (W_t)- are used for the characterization of grindability at the moment. Generally the grindability tests are carried out under simplified circumstances, in special dry batch mills, with defined feed size material, until prescribed grinding fineness at room temperature.

However, it can be seen in such a case in the industrial practice, where the conditions are essentially divergent from that of the above mentioned. For instance, grinding is frequently performed in water, liquor or acid and often at high temperature (e.g. bauxite grinding during Bayer-process, ore grinding in hydrometallurgy).

Nowadays, the fine- and ultra fine grinding (nano-grinding) is a common task, when a fine material (< 50…200 µm) has to be ground down to the fineness of few micrometers or nanometers. The traditional, present grindability test methods (Bond-, Hardgrove-, Zeisel-processes) are not suitable directly, since according to these prescriptions the maximal particle size of feed material (0,5…..3 mm) is bigger than that of the examined sample.

There are traditions of grinding- and grindability experimental investigation at the University of Miskolc, Institute of Raw Material Preparation and Environmental Processing (previously called Department of Process Engineering): professor Gusztáv Tarján dealt with the processes placed in ball mills, the movement of grinding media [3, 4]. Professor Szilveszter Pethő conducted research into modeling of comminution and classification, the question of computer controlled processes [5, 6]. Professor Barnabás Csőke dealt with mathematical modeling of crushing and grinding, the dimensioning of mills and the testing of grindability

[1, 6, 7]. I joined in this research work to investigate the above mentioned field in 2002 as full time PhD student.

Among the Hungarian scientific school working in the field of comminution the SZIKKTI, CEMKUT and University of Veszprém have to be emphasized beside University of Miskolc, where professor Béla Beke [8, 9, 10] and Ludmilla Opoczky, professor Zoltán Juhász A., furthermore Sándor Verdes scientists have been working.

Based on the survey of literature the following conclusions were drawn:

Literature does not deal adequately with experimental investigation of grindability conducted under special circumstances – e.g. high temperature, alkaline media, extra fine feed particle size. On the one hand it can be explained by the lack of a universal theory for particle size – grinding energy relationship, on the other hand the presently used apparatuses and methods are not suitable for investigation of grindability under different special conditions.

II. Aims of research

The main aim of my research was to develop such grindability testing apparatuses and methods which can be applied for:

- determination of grindability in the field of ore- and mineral mining, mineral processing, cement industry and in the waste treatment as well;
- grindability testing under special grinding circumstances, i.e. in aqueous, alkaline or acidic media, at temperature between 20…200 ^{o}C;
- fulfillment of grindability test of brittle materials with diverse and arbitrary feed particle size;
- determination of specific grinding energy, which can be successfully used in designing of new grinding facilities, and in optimization of the existing operations (grinding ball charge, filling ratio, operating of classifier,…), resulting in significant savings in both investment and operating costs

One of the important goals is that the developed grindability testing methods have to be suitable for fast determination of grindability, in this way methods can be used in the industrial practice for controlling grinding process.

III. Apparatuses, testing- and evaluation methods

The research work consists of survey of Hungarian and international literature, developing of experimental apparatuses and -methods for grindability testing, laboratory and industrial scale experiments, evaluation of results and drawing of conclusions.

Most of my scientific results are based on systematic laboratory experiments carried out at the University of Miskolc, Institute of Raw Material Preparation and Environmental Processing. These laboratory results were compared with industrial scale data to prove their practical applicability.

Developed apparatuses and applied equipments

After elaborating the literature the main task was to develop the testing apparatuses. I took part in the modification of Hardgrove-mill, the upgrade of Bond-mill was performed under my professional guidance. Both mills were adapted to be heated and were equipped with devices suitable for measuring of electric power and grinding work.

Fig.1: Modified Bond-mill

Fig. 2: "Universal" Hardgrove-mill

Modified Bond-mill

The electric heating of drum mill was realized by 3000 W to obtain a homogenous temperature distribution in the grinding chamber, several heating wires have to be installed. Due to the 3 kW heating wires the temperature can be adjusted from 20 up to 200^{o}C easily and quickly with a tolerance of ± 1ºC. Microprocessor-controlled KD24D type universal instrument and Ni-CrNi thermocouple were installed for controlling and measuring the temperature.

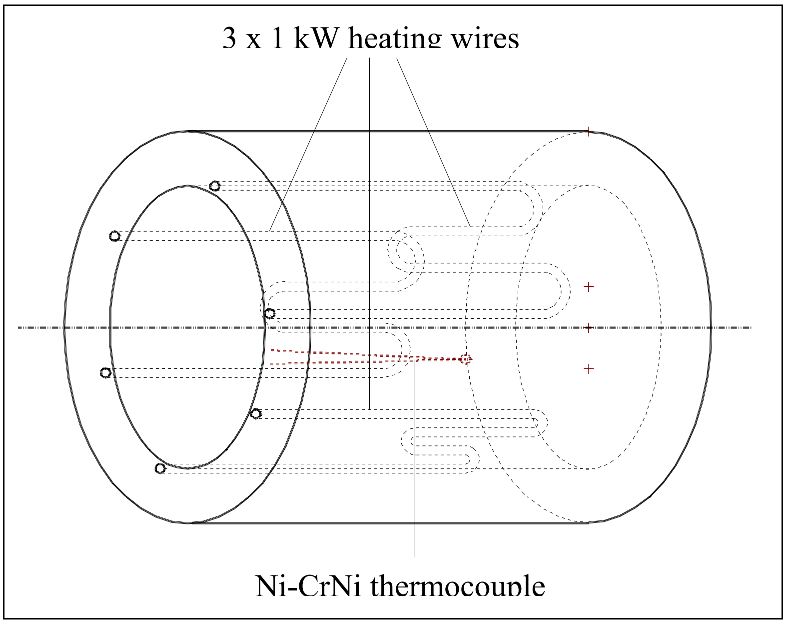

Fig. 3: Schematic figure of mill heating

The used electric power has been measured by microcomputer controlled digital energy meter Carlo Gavazzi WM1-DIN during the experiments for the characterization of grindability. Beside momentary power the work used for grinding, the current (I), the voltage (U) and cosφ can be measured as well by this device.

Universal Hardgrove-mill

The required constant temperature was reached due to the heatable jacket (Fig.4) around the grinding chamber which can be adjusted in the temperature range of 20 … 300 ºC (±1 ºC).
For measuring the temperature 4 pieces of PT100 platinum thermocouples were built in. The heating process is controlled by a PC program (written by Dr. József Faitli).

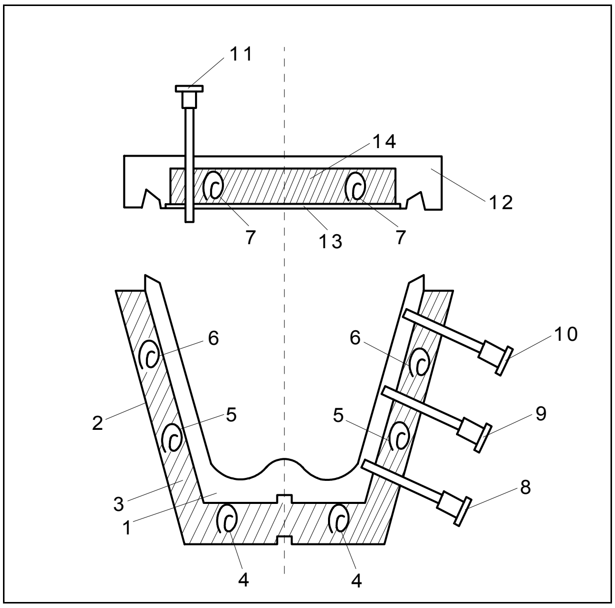

Fig.4: Schematic figure of heatable grinding chamber

The mill was equipped with a simple torque-meter (load cell) which enables the power delivered to the grinding chamber to be measured directly. For this reason the whole grinding chamber (1) is mounted on an axial bearing (5), so it can rotate practically freely. This rotation is fixed by a force arm (2) connected into a force transducer (4). By this way the torque necessary for the grinding can be measured.

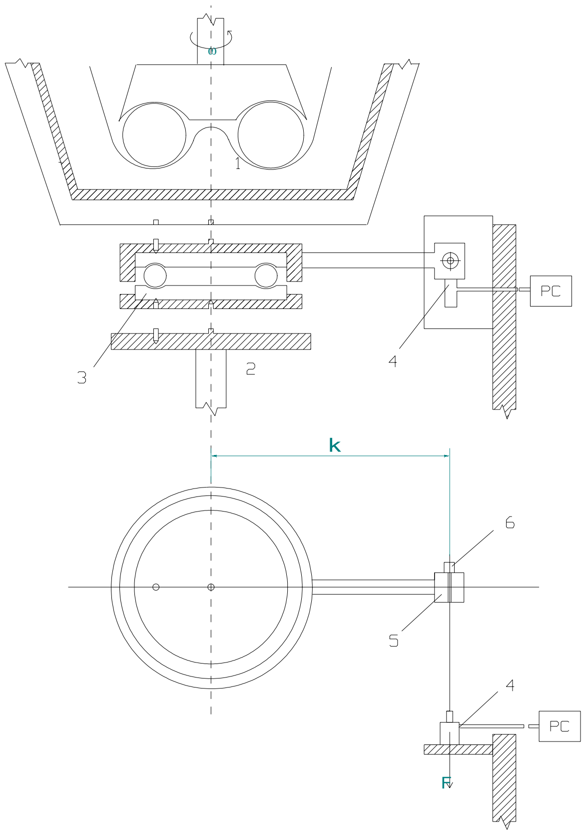

Fig.5: Torque measuring device

The exact determination of particle size distribution is a fundamental point of experimental testing. To this end standard wet sieving (ISO-3310.1 sieves) and Fritsch Analysette 22 type wet laser granulometer was used.

Experimental

Important goal of my dissertation was to develop a proper measuring- and evaluating method for both Bond- and Hardgrove-mills. Furthermore a comparative investigation was carried out and relationship was created between them (grindability numbers).

Therefore different samples were selected for experiments. Bauxite, limestone, pebble, basalt, zeolite, fly ash and alumina were used as model materials and the applicability of new methods as well as apparatuses were proved through numerous measurements.

The research and development work was conducted in three working stages:

1. Firstly, the laboratory conditions of high temperature alkaline grinding were created for the investigation of bauxite's grindability at high temperature. To this end both mills were equipped with lagged heated jacket. In this measuring series the grindability of karst- and lateritic bauxite with different texture (boehmitic and diasporic Greek, boehmitic Hungarian from Halimba, gibbsitic Australian from Weipa, boehmitic African from Boké) was measured with modified Bond- and Hardgrove-mill parallel at different temperature. Additionally, the question of grinding

aid was examined. In this case bauxite sample with desired particle size was added into the mill. Evaluation was performed according to "traditional" way.

2. In the second working period the Hardgrove-mill was equipped with a "MOM Kaliber 8964" type load cell, the Bond-mill was upgraded with a "Carlo Gavazzi WM1-DIN" type electric power measurer. Measuring and evaluation method was worked up and the applicability of them was proved. In this case limestone, pebble, basalt, zeolite samples were used for experiments in both mills parallel. The feed particle size and other circumstances were as traditional ones, but during the grinding experiment the input work was measured directly. This series proved that the determination of Bond- and Hardgrove-indices originate in the direct measuring of grinding work.

3. In the third period, the developed apparatuses, measuring- and evaluating methods were applied for the investigation of "real" industrial tasks:
   a) for characterization of grindability of fine particulate materials, i.e. alumina and fly ash;
   b) for the grindability measurement of mixtures (composite cements, separate- or intergrinding?);
   c) the laboratory results were compared with data originated from industrial measurements:
      - with grinding experimental data of laboratory – pilot plant scale stirred ball mill;
      - with parameters of industrial measurement, which were carried out with different throughput ball mills (4 t/h and 100 t/h) in the plant of MAL Zrt. (Ajka) and Holcim Zrt. (Miskolc - Hejőcsaba).

The elaborated testing- and measuring methods were applied successfully in numerous industrial research works.

Evaluation of the results

The measured data were processed in tables and diagrams, the evaluation was fulfilled by „temperature - Bond-work index" diagram in the first case and by „ground material particle size – energy" relation in the second case. Furthermore the well-known methods of process engineering (distribution functions), mathematical statistics and correlation counting were used for evaluating. For description of the processes different functions were fit into the measured points and the value of correlation coefficient, absolute or relative error were given.

IV. Scientific achievements, theses

The new scientific results of my dissertation can be summarized as follows:

1. The „universal" Bond-mill and the relevant testing method were developed based on direct energy – electric power – measurement. This method is appropriate for the determination of material's grindability.

During the experiment the grinding work is measured in the steady state, from which and knowing P_0 no-load power the grinding work for one revolution is:

$$W^* = \frac{\int (P(t) - P_0)dt}{N} \qquad (1)$$

the specific grinding work for one revolution:

$$W_f^* = \frac{W^*}{G}. \qquad (2)$$

The Bond-work index can be calculated using by:

$$W_{iB} = \frac{W_f^*}{\left(\frac{1}{\sqrt{x'_{80}}} - \frac{1}{\sqrt{X'_{80}}}\right)} \qquad (3)$$

formula, where X'_{80} and x'_{80} are 80 % passing size of the feed and the ground material [μm], G is the grindability factor [g/revolution], viz. the weight of „fresh" <100 μm product produced in the laboratory mill through one revolution in the steady state, N is the revolution number in steady state.

2. The testing Bond-mill equipment and testing method was improved to determine the grindability at high temperature (in the range of 20…200 °C) in alkaline medium.

3. The „universal" Hardgrove-mill and the relevant testing method were developed based on direct energy – grinding torque – measurement. This method is appropriate for the determination of material's grindability.

Pending the test the force F(t) (torque) influenced on the force arm is measured from which:

$$M(t) = k\,F(t) \qquad (4)$$

the specific grinding work (knowing no-load torque M_0) can be calculated:

$$W_h = \frac{\int_0^{\tau} \{2\pi n[M(t) - M_0]\} dt}{m_p} \tag{5}$$

where m_p is the mass of finished product, the mass of particles smaller than 75 µm in the mill product,
the Bond-work index is:

$$W_{iB} = \frac{W_h}{\left(\frac{1}{\sqrt{x'_{80}}} - \frac{1}{\sqrt{X'_{80}}} \right)} \tag{6}$$

where x_{80} is the 80 % passing size of finished product (<75 µm) and X_{80} is the same size of the feed.

4. The Hardgrove -mill testing method was improved to determine the grindability at high temperature (in the range of 20…300 °C) in alkaline medium.

5. It was proved that the Csőke-formula

$$W_{iB} = \frac{468}{HGI^{0,82}} \tag{7}$$

is appropriate for calculation of the Bond-work index from the Hardgrove grindability index determined using by the developed „universal” testing and evaluation method.

6. Based on the experiments with **fine feed particle size materials** (e.g. alumina, power station fly ash, x_{max}**<100 µm**) it was established that the **grinding performed in the „universal” Hardgrove-mill simulates the grinding circumstances (mechanical stress) of pilot plant scale stirred ball mill and industrial scale drum ball mill more accurately than the grinding in „universal” Bond-mill**. (Median size of final product is 3…10 µm)

Namely the friction (abrasive) work is dominant in the “universal” Hardgrove-mill as well as in the stirred ball mill and in the traditional ball mill. It can be seen in the Fig. 6., where the relation between specific grinding work W_s and median of finished product x_{50} is described.

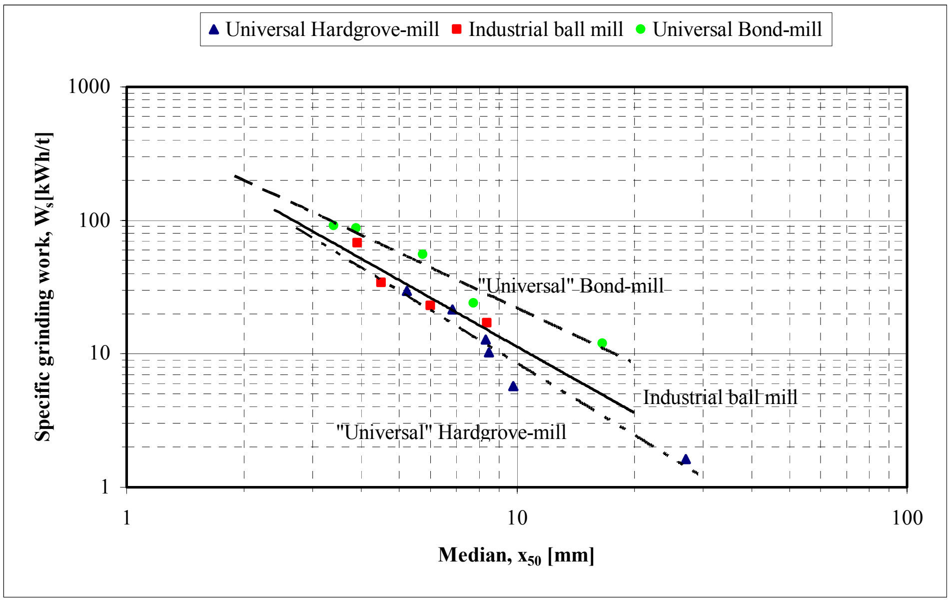

Fig. 6: Relation between specific grinding work W_s and median size of finished product x_{50} in case of alumina

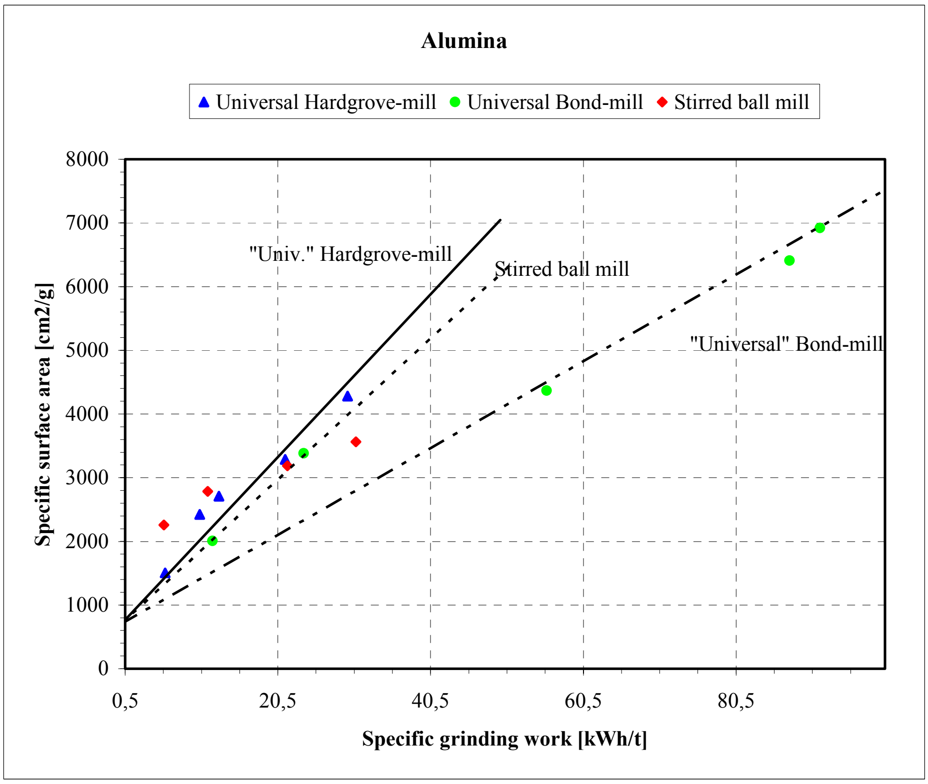

Fig. 7: Relation between specific grinding work W_s and specific surface area of finished product S in case of alumina

6.1 It can be concluded from Fig. 6 and 7., that the „Universal" Hardgrove mill is more suitable for characterization of grindability of fine materials than „Universal" Bond mill.

Therewith the values obtained from „Universal" Hardgrove mill method are need to be corrected according to following formula:

$$W_{fajl}^{keverömalom} = kW_{fajl}^{mért} \qquad (8)$$

The correction factor of Eq. 8 is **k** = 1/m = 1/0,7869 **=1,27**.

7. Based on the experimentally obtained grindability values for **bauxites of diverse mineral composition and texture** in **alkaline media at various temperature** it was established that the **grindability** of examined materials has **improved** at a high temperature and in an alkaline medium. The specific grinding energy decreased. Nevertheless, the degree of the improvement was not adequate for different bauxites.

7.1. A significant improvement of grindability value occurred in the case of bauxite samples of autogenous texture type (oolitic, pisolitic Boké and Weipa, oolitic Halimba) in alkaline medium at temperature 20 °C compared with the value characteristic for dry grinding. The degree of improvement increased at 60 °C. At the same time the grindability of pelitomorf Greek bauxites improved only slightly in an alkaline at 60 °C (Fig. 8).

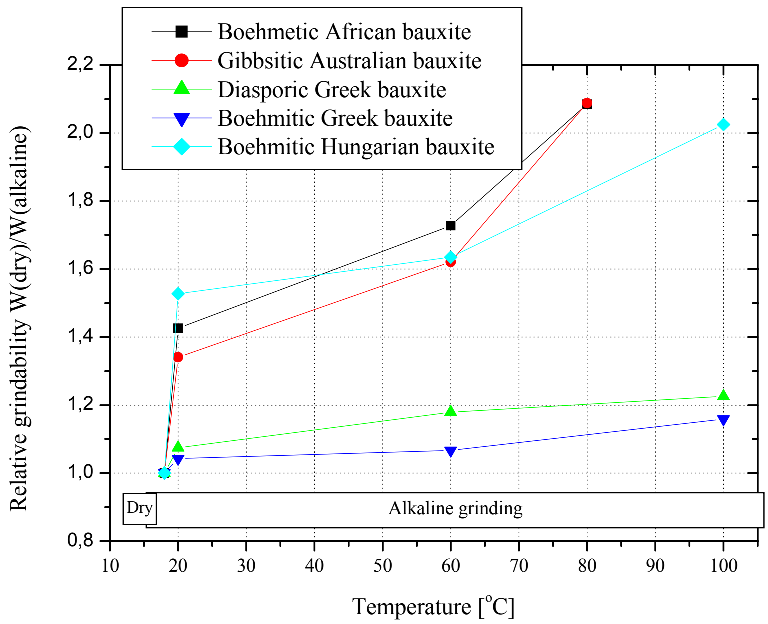

Fig. 8: Changing of bauxite's grindability as function of temperature

The changing of grindability can be taken into account with the correction

$$W_{iB,korr} = \frac{W_{iB,száraz}}{k_i} \qquad (2)$$

of Bond-work index determined under dry condition, where „k" (temperature and bauxite type dependant) is the relative grindability-changing correction factor.

7.2. Investigating the effect of CaO and $Ca(OH)_2$ on the bauxite's grindability it was stated that:
The Bond-work index of bauxite decreased using by both grinding aid but the degree of changing was different depending on bauxite type:

- in case of diasporic Greek bauxite after an intensive grindability index decreasing effect of CaO the forms of W(T) grindability – temperature curves are almost the same;

- in case of boehmitic Greek bauxite with higher caolinite content the effect of CaO on the grindability improvement intensify as function of temperature (reaction of caolinite and CaO);

- it can not be observed significant effect of grinding aid on grindability of the non-cohesive relatively good quality Hungarian (lower caolinite content) boehmitic bauxite;

- furthermore the different grinding aid (CaO and $Ca(OH)_2$) diminished the Bond-work index of boehmitic and diasporic bauxites in different degree as function of temperature.

8. Based on the experimental results it was proved that the values determined using the developed „universal" grindability test based on direct energy measurement and the relevant evaluating methods can be applied and implemented in the industrial practice using by an appropriate „conversion factor".

Additionally, it was demonstrated that the value of the conversion factor depends on the testing method and relevant mill (Hardgrove, Bond), as well as on the texture of material being tested.

8.1. Based on the good correlation between methods and on the diagram containing all measured data it can be stated that only a slight value of conversion factor is needed:

$$W_{i,B}^{traditional} = 0{,}97 W_{i,B}^{energy\_measurement} \qquad (9)$$

8.2. In case of Hardgrove-mill a lower rate of conversion factor between traditional and energy measuring methods is necessary:

$$W_{i,B}^{H,traditional} = 1{,}02 W_{i,B}^{H,torque\_measurement} \qquad (10)$$

8.3. The so called "industrial" Bond-work index originated from „universal" Bond-method based on energy measurement is higher than that of the traditional one, which requires higher rate of conversion factor:

$$W_{i,B}^{B,traditional} = 0{,}88 W_{i,B}^{energy\_measurement} \qquad (11)$$

8.4. The rate of conversion factor depends on the examined mineral. The relative deviation of Bond-work indices originated from energy measuring Bond- and Hardgrove-method in the case of inhomogeneous texture bauxite and zeolite is higher (+18,58 %, -19,5 %), and in the case of homogenous texture limestone, crushed pebble and basalt is lower.

V. Opportunities for application

The developed „universal” Hardgrove- and Bond-mills as well as grindability testing methods – which support to determine the grindability of brittle materials under different circumstances (aqueous, alkaline and acidic media and at high temperature) - can be utilized in several industrial field in a wide range.

For example the „universal” Hardgrove-mill can be applied for fast and regular grindability testing in industrial grinding plant and in this way it can be used for controlling of the mills. Namely the changing of grindability of material being ground affects the operation of mills and its controlling.

The grindability numbers determined using by „universal” Hardgrove- and Bond-mills can be applied in case of dimensioning of mill, determination of operational parameters (size distribution of grinding media, ball filling ratio, operation of classifier, etc…) as well.

My research work is close connected to the projects of the Institute of Raw Material Preparation and Environmental Processing in the last six years. The developed grindability testing methods were applied successfully in several industrial research tasks for determination of specific grinding energy and Bond-work index. The results of two of these tasks – industrial scale ball mill and ring mill - have to be emphasized.

Since recently the cement industry show an interest in my research results as well, during my further work it is scheduled to improve the developed „universal” grindability testing methods to be suitable for precise determination of grindability of reduced clinker factor – composite and composite Portland – cement. This is important from the technical point of view and because the quantity of so called reduced clinker factor cements – containing one or more cement complement material - has been increased all over the World and in Hungary as well. This is related to one of our actual environment protection problems in these days, the reduction of CO_2 emission connected the climate changing.

VI. Literature

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2. Csőke, B., Mucsi, G., Opoczky, L., Gável, V.: **Modifying the hydraulic activity of power station fly ash by grinding (Beeinflussung der hydraulischen Aktivität von Kraftwerksflugasche durch Mahlung)** Cement International No. 6/2007. Vol. 5. ISSN 1610-6199 pp. 86-93.

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5. Csőke, B., Mucsi, G., Opoczky, L., Gável, V.: **Modification of fly ash properties by grinding.** 11th Symposium on Comminution, 2006. Budapest, Hungary (CD-ROM) ISBN 963 9319 589

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7. Csőke, B., Mucsi, G., Csende, L., Balogh, Z.: **Quality Control For the Production of Alumina Grinds Using Ball Mills.** XXIII. Mineral Processing Congress, 2006. Istanbul, Turkey (Proceedings), (Ed. Önal et al.) Promed.Ad.Ageincy, pp. 121-126. ISBN 975-7946-27

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