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Influence of High Pressure Grinding Rolls on Physical Properties and Impact Breakage Behavior of Coarsely-sized Cement Clinker

Mahmut Camalan

Department of Mining Engineering, Middle East Technical University (METU), Ankara, Turkey

Mehmet Ali Recai Önal*

Department of Metallurgy and Materials Engineering (MTM), University of Leuven (KU Leuven), Heverlee, Belgium

*Address correspondence to Mehmet Ali Recai Önal. Tele: +32 016321780. E-mail: mehmetalirecai.onal@mtm.kuleuven.be

Abstract

High pressure grinding rolls (HPGR) are widely used in cement clinker grinding prior to ball milling. The efficiency of HPGR was previously related to two capabilities: (a) efficient stressing mechanism, (b) weakening of particles which lead to finer product sizes after subsequent ball milling. This study aimed to investigate the influence of HPGR on the impact breakage behaviour of coarsely-sized cement clinker. Drop weight tests were conducted on three single-size samples from both feed and product streams of an industrial HPGR, so as to obtain impact breakage parameters: breakage probability and impact breakage functions. Radiographic, Vickers micro-hardness, BET, SEM, XRD, XRF and image processing methods were utilized to explain the variations in impact breakage parameters. The product samples were found to have less porosity

than feed samples but they contained HPGR-induced micro-cracks. Those cracks are believed to be responsible for lower hardness and higher breakage probability of product particles than of feed ones under impact. However, fragments generated from the product samples by drop weight tests were coarser than those generated from feed samples. That was due to elimination of pores by HPGR action. At excess energy levels, both samples were broken to same extent regardless of structural differences.

Keywords: drop weight test, fragmentation, high pressure grinding rolls, impact breakage parameters, portland cement clinker

INTRODUCTION

Increasing energy consumption in comminution requires the development of new comminution equipment. A recently-developed equipment to serve this purpose is the high pressure grinding rolls (HPGR) which is commonly adopted to cement grinding circuits. HPGR consist of a pair of rotating rolls through which a bed of particles are nipped and ground with high external pressure exerted on the particle bed (Schönert 1988). In cement clinker grinding, HPGR have recently been used prior to ball mills so as to decrease energy consumption in conventional ball mill alignments. Patzelt (1992) stated that certain HPGR-ball mill arrangements consume 30% less energy than those with only ball mills. This is supplemented with case studies from cement manufacture plants adopted HPGR (Von Seebach, Neumann, and Lohnherr 1996).

Researchers linked the energy-efficient use of HPGR-ball mill arrangements with two main reasons. When low degrees of size reduction ratio was considered, HPGR was found to consume lower energy due to its compressive loading mechanism compared to its counterpart (Fuerstenau et al. 1990; Fuerstenau and Vazquez-Favela 1997). This advantage has strengthened the use of

HPGR as a preferred pre-grinder before ball mill operation. The other main reason for high efficiency of HPGR equipment was correlated with the external stresses applied by the rolls on the particle bed. Several researchers concluded the effect of these external stresses during HPGR operation as damaging of the particles at various degrees by high inter-particle stresses that induce micro-cracks in particles (Kodali et al. 2011; Ozcan and Benzer 2013; Patzelt, Knecht, and Baum 1995; Ghorbani et al. 2011). In subsequent ball milling, these damaged particles tend to grind from a certain feed size to a certain product size with lower energy requirement due to these micro-cracks (De 1995; Fuerstenau and Vazquez-Favela 1997; Fuerstenau, Lutch, and De 1999). Some researchers additionally emphasized that there is a heterogeneity in ball-mill breakage characteristics of a HPGR product due to damaging effect of rolls on a certain proportion of the feed material (De 1995; Fuerstenau, De, and Kapur 2004; Camalan and Hoşten 2013).

Although numerous studies were performed on the effect of HPGR on subsequent ball mill breakage properties, few contained the effect of HPGR operation on the breakage properties of coarsely sized particles. The reason was probably that the energy load given by batch ball mill was quite insufficient to fracture coarse particles (Austin, Klimpel, and Luckie 1984). In this case, single-particle breakage methods can be one of the solutions to this problem where particles are stressed one by one. Indeed, Tavares (2005) utilized from this method to compare the impact fracture energies of coarsely-sized HPGR and conventional crusher products. This study showed that the lower mean-fracture energies of HPGR product with respect to conventional crusher products could have indicated a direct evidence for the weakening effect of HPGR operation.

The aim of this study is to investigate the effect of HPGR on the impact breakage parameters of coarsely-sized cement clinkers by examining samples from both feed and product streams of an industrial production line. For that purpose, drop weight test was performed which

has been used for single-particle breakage of various material types including most of the minerals (Napier-Munn et al. 1996; Bearman, Briggs, and Kojovic 1997; Kapur, Pande, and Fuerstenau 1997; Avşar 2003; Genç, Ergün, and Benzer 2004; Shi and Kojovic 2007; De Souza et al. 2008; Rizmanoski 2011). In order to explain the variations in impact parameters of both sample types, several methods were conducted to analyse them by means of their chemical, physical and mechanical properties.

Single-particle Breakage Tests

Single-particle breakage tests give an insight for understanding breakage process in micro-scale events. The tests are based on stressing particles one by one either with a certain type of loading mechanism including impact, compression or shear loading with auxiliary stressing equipment; or throwing the particle with a certain impact velocity onto a plate. Various loading equipment for single-particle breakage can be found in detail elsewhere (Tavares 2007).

There are three types of data obtainable from single-particle breakage: (i) functional relationship between stressing energy and corresponding product size distribution (Napier-Munn et al. 1996; Petukhov and Kalman 2003; Vogel and Peukert 2005; Meier et al. 2009; Rozenblat et al. 2012), (ii) probability of particle breakage as a function of stressing energy (Krogh 1980; Austin 2002; Cho and Austin 2003; Vogel and Peukert 2003; Antonyuk et al. 2005; Aman, Tomas, and Kalman 2010) and (iii) specific fracture energy or fracture strength distribution of a given material (Bourgeois 1993; Tavares and King 1998).

Breakage Parameters of the Single-particle Breakage Test

Similar to those in the batch ball mill, two main breakage parameters can be defined from the single-particle breakage test: (a) breakage probability and (b) impact breakage function. The

former is the cumulative mass of the single-size particles broken under a given E_{is} and pass through a certain size of a sieve. The relationship between breakage probability and E_{is} is analogous to the concept of breakage rate in ball milling. It gives a probability upon which a particle will be broken after applied impact energy. However, breakage probability should not be confused with the term ‘fracture probability’ which is the probability that a fracture will be initiated at a certain applied energy (Tuzcu and Rajamani 2011).

On the other side, impact breakage function can be described as the cumulative size distribution of progeny fragments produced after a certain E_{is} . It can be calculated by Eq. 1.

$$B_{i1} = P_i / P_2, \text{ where } i > 1 \quad (1)$$

B_{i1} represents the cumulative mass fraction of material broken from single-size interval indexed as ‘1’ which appears in the size intervals less than the upper sieve of the size interval ‘i’. P_i and P_2 are the cumulative mass percentages of materials passing from the upper sieve of the size interval ‘i’ and ‘2’, respectively, after drop weight test.

Structural Discontinuities and Their Effect on the Breakage Behaviour of Brittle Particles

Discontinuous structure of clinker particles could have a significant effect on the impact breakage of the clinker. Several studies regarding the effect of the particle discontinuities on fragmentation along with mechanical and bulk properties of rocks and other materials can be found in the literature (Brown, Biddulph, and Wilcox 1964; Bevilacqua and Ferrara 1996; Walsh 1966; Boggs 1967; Griffiths, Davies, and Bassett 1979; Subero and Ghadiri 2001; Tavares and Cerqueira 2006; De Souza et al. 2008; Tavares and Dasneves 2008). The definition of discontinuities (i.e., porosity) involves only pores or only cracks or a mixture of these two defect types. Not only the

degree of porosity but also the shape and distribution of its constituents (i.e. pores and cracks) have equally important effects on the mechanical and bulk properties of the materials (Brown, Biddulph, and Wilcox 1964; Walsh 1966). It is widely accepted that these two defect types generally decrease the stress-bearing capacity of brittle and polycrystalline materials (e.g. ceramics and rocks) by contributing to intensification of stress concentrations causing unexpected failures or premature complete breakages of such materials (Griffiths, Davies, and Bassett 1979; Paterson and Wong 2005). Bevilacqua and Ferrara (1996) quantitatively showed that in a porous material, comminution behavior of coarse particles larger than the mean voids-inter-space is related to the bulk mechanical properties of porous material, whereas the comminution of the very fine material below this interspace is related to the mechanical properties of the non-porous material (i.e. the matrix). Thus, a transitional region between the coarse and the fine particle sizes appear (representing grains with around the mean void interspace size) in the size distribution of comminution products thereby giving bi- or trimodal distributions. The clinker and individual clinker constituents mostly show elastic or viscoelastic deformation under force-displacement curves having high modulus of elasticity values (Tavares and Cerqueira 2006; De Souza et al. 2008). Hence, it is fair to estimate that clinker particles will also experience brittle failure under external stress. Consequently, structural discontinuities within the clinkers were also included as an important factor within this study which can affect the impact breakage behaviour.

MATERIAL AND METHODS

The experimental material composed of three narrowly-sized samples ($-25.4 + 19.0$ mm, $-19.0 + 12.7$ mm and $-12.7 + 9.53$ mm) of Portland cement clinker. The samples were extracted from the feed and product streams of an open-circuit, industrial-scale HPGR (Bastas Cement Inc.)

operating at 10 MPa pressure with 250 tons/hour capacity. The particle size distributions of both streams are given in **Figure 1**. The HPGR has a roll diameter of 0.8 m and a roll length of 1.0 m while the gap distance between rolls is 15 mm. De-agglomeration of product samples were omitted since no flakes were observed in the tested size fractions. Chemical analyses of each single-size level of feed and product classes were performed by Spectro IQ model X-ray fluorescence (XRF) spectrometer. Cylindrical pellets were prepared for XRF analyses. The mineralogical characterization of both sample classes were performed on $-12.7 + 9.53$ mm single-size samples with Rigaku Ultima-IV model X-ray diffractometer (XRD) with a $\text{Cu-K}\alpha$ X-ray tube operating at 40 kV and 40 mA.

Drop-weight Test

Drop weight test is a special sub-type of single-particle breakage test. This test consists of stressing particles one by one by dropping a cylindrical or spherical steel mass from a noted height. The specific impact energy (kWh/t) exerted on a set of single-size particles can be determined by adjusting drop weight and drop height in Eq.2.

$$E_{is} = \left[(2.78 \cdot 10^{-4}) * M * g * (h_0 - h_f) \right] / \bar{m} \quad (2)$$

Here, E_{is} is the applied specific impact energy (kWh/t), M is the mass of the drop weight (kg), g is the gravitational acceleration (m/s^2) taken as 9.81, h_0 is the initial drop height (m) measured from the surface of anvil to the bottom of drop head, h_f is the offset height (m) between the bottom of the drop weight and surface of the anvil after impacting on the particle, $\bar{m}$ is the average weight of a particle in the set of tested particles (kg), and the term “ $2.78 \cdot 10^{-4}$ ” is the conversion factor to equate the unit of E_{is} to kWh/t.

The three coarsely-sized samples of HPGR feed and product classes were subjected to 5–7 impact energy levels. The drop weights were either 1.24–2 kg, whereas a height range between 0.04 m and 1 m were studied. Lower drop-heights, i.e. lower impact energies, were not possible to study due to equipment restrictions. The applied specific impact energy levels for each tested size level of these samples are summarized in **Table 1**. Each tested sample contained 50–200 particles. The particles produced by the drop weight tests were subsequently subjected to dry screening for determining their size distribution. All obtained results from each single-size level of HPGR feed and product classes were compared for the same applied specific impact energy level.

An important aspect of the drop weight test is that the affinity for fragmentation can be estimated by fitting an empirical relationship between a degree of product fineness and specific impact energy or E_{is} (kWh/t). This degree of fineness is defined as t_{10} , which is the cumulative mass percentage of the product passing 1/10th of the original feed size. Then, the relationship between specific impact energy and t_{10} for a given narrowly-sized sample might be fitted into a functional form as in Eq. 3 (Napier-Munn et al. 1996).

$$t_{10} = A * [1 - \exp(-b * E_{is})] \quad (3)$$

Here, A and b are functional parameters to be estimated. These values in Eq. 3 can be found by fitting the t_{10} - E_{is} data to the equation for each size-level of HPGR feed and product samples. In order to achieve the estimates of A and b , non-linear least squares regression analysis was used. The product of the parameters A and b is the slope of the E_{is} - t_{10} curve at zero input energy. This value can be used to characterize the amenability of the particles for fragmentation (Tavares 2007). The lower values of $A*b$ indicate higher resistance against fragmentation whereas higher values indicate more amenability for fine fragmentation.

Determination of Structural Discontinuities

Four characterization methods were used to directly determine the degree, distribution, shape and roundness of bulk and/or superficial discontinuities: radiography, Vickers micro-hardness, brunauer-emmett-teller (BET) and scanning electron microscopy (SEM) with image processing CLEMEX© software.

Radiographic Testing

Radiographic testing is widely used as a non-destructive testing method for visualization of surficial and bulk states of materials. In this method, selected particles are located between a short-wavelength X-ray source and X-ray sensitive film. Generated X-rays penetrate into particles and relatively pass to the placed film creating an invisible image. After the test, this film is treated with a specific salt solution in the dark chamber for a certain time interval to have a final image that is visible under halogen/fluorescent/LED lamps. In such a radiographic film, flaw regions (e.g. cracks, pores) will create darker traces since X-rays are much less adsorbed or scattered by flaws thereby exposing the film. Solid regions are denser than flaw regions meaning that X-rays are highly absorbed or scattered in those regions thereby reducing the intensity of exiting radiation. The more intact is a region, the less of amount of X-rays with less intensity will make to the film and hence the brighter will be the trace of the region on the film. For this study, cloudy darker regions were commented as porous regions while a linear dark trace were defined as a macro-crack. Regarding the average size of the tested particles in this study, the detectable discontinuities in the radiographic results could be considered only at macro-scale and does not necessarily mean the absence of micro-cracks.

Radiographic tests were conducted on randomly taken feed and product samples from both $-19 + 12.7$ mm and $-12.7 + 9.53$ mm single-size levels. Seifert 200 kV radioscopy device operating at 80 kV and 5 mA was used as X-ray source while the particles were distinctively placed on Agfa Structurix D4 type films prior to testing. After each testing, the film was placed on a LED-illuminated flat table with constant illumination parameters, and a digital image was taken with a standard camera giving a resolution of 72 dpi (both vertical and horizontal) image.

SEM and Image Processing Analyses

SEM examinations were applied to obtain several secondary electron images of $-12.7 + 9.53$ mm feed and product samples by using Fei Nova Nano 430 model device. Several secondary electron images were selected and manually cropped to satisfy the working principles of processing software trademarked as CLEMEX©. This software is basically designed as a binary-image processor where individual pixels in a grey-scale image (such as in SEM images) are grouped, coloured and processed with respect to their position on a software-defined grey-colour histogram. In this study, pixels in the SEM image below a certain threshold were qualitatively assigned and colored as darker regions by the software. By doing so, those darker regions were obviously defined as porosities on the examined particles.

Several porosity parameters for each processed SEM images can be calculated by this software including sphericity, roundness and mean spherical diameter. Sphericity, by definition, is the estimation on the relative shape of an object with respect to a sphere. Roundness, not to be confused with sphericity, is a shape measure that quantifies the degree of sharpness of the edges and corners of an object (Boggs 1967). Equivalent spherical diameter given by the program is the diameter of any three-dimensional sphere having the same surface area of the two-dimensional,

cross-sectional area of object measured in the image. The respective formulas to calculate these parameters were provided by the analysis software and are given by Eqs. 4–6.

$$Sphericity = 4 * \pi * Area / (Perimeter)^2 \quad (4)$$

$$Spherical \ Diameter = 2 * (1.2247) * (Area / \pi)^{1/2} \quad (5)$$

$$Roundness = [4 * (Area)] / [\pi * (Length \ of \ long \ axis)^2] \quad (6)$$

The more spherical or round is an object, the closer the sphericity or roundness value will get to 1 on a 0 to 1 scale where unity refers to perfect sphere or perfect roundness. An object could have high sphericity but low roundness at the same time or vice versa. The mean, standard deviation as well as the minimum and the maximum values of each of these parameters can also be calculated by the software for each examined image.

BET Analysis for Porosity Measurement

BET analysis was applied to determine the specific surface area of and total pore volume in $-19.0 + 12.7$ mm feed and product particles. This method consists of calculating the amount of nitrogen adsorbate to yield a monomolecular layer on the sample surface simply through adsorption isotherms which describe functional relationship between pressure and volume of the adsorbed gas at a constant temperature (Braunauer, Emmett, and Teller 1938). The required adsorption-desorption isotherms were taken from AUTOSORB-6B surface characterization equipment. For all BET analyses within this study, the monolayer adsorbate coverage occurred at a low relative pressure. This pressure is defined as the ratio of absolute pressure of nitrogen to its normal vapour pressure and was around 0.31. The adsorbate coverage was used to estimate specific surface area. Determination of pore volume, on the other hand, was calculated from the adsorbed

nitrogen volume at a relative pressure of nearly 0.96. The calculation procedure for the pore volume along with pore size distribution are given by Barrett, Joyner, and Halenda (1951).

Vickers Micro-hardness Tests

The Vickers micro-hardness tests were performed by 9.81 *N* applied force for 10–15 seconds in order to determine and compare the micro-hardness values of both sample classes. three particles of feed and three particles of product samples were randomly taken from –19 + 12.7 mm single-size level. The particles were grouped with respect to their type, cut and mounted into two polished sections. Twenty safely separated indentations were taken from each polished section. The impression diagonal length in all hardness tests varies between 65–85 μm .

RESULTS AND DISCUSSION

Results

The chemical analyses results are given in **Table 2** for both HPGR feed and product samples at all single-size levels. Alumina modulus (AR), silica ratio (SR) and lime saturation factor (LSF) were also calculated for both sample types with their corresponding formula (Jackson 2003). The results showed that chemical analyses were more or less identical for each size fractions of both sample classes. In **Figure 2**, XRD patterns for HPGR feed and product samples with –12.7 + 9.53 mm single-size level are given. The major phases were found as alite and belite with lesser amounts of C3A and C4AF. In minor amounts, calcium sulphate was also detected. Resultantly, both product and feed samples had exactly the same mineralogical compositions. Accordingly, chemical and mineralogical states of product and feed samples were eliminated as

constants and were not considered as possible factors that can cause a difference in impact breakage properties of these samples.

In **Figure 3**, the breakage probabilities of each size-level are given for both HPGR feed and product samples. Additional two tests were done for reproducibility of breakage probabilities of $-19 + 12.7$ mm feed and product samples under the same 0.44 kWh/t impact energy level. Based on these results, the variations of the breakage probabilities of both feed and product samples were found as equivalently 3% from the original points in **Figure 3**. Up to a certain level of the applied impact energy, the breakage probability values were higher for product samples than for feed samples in all single-size levels. Hence, these results show that HPGR feed samples of each size class were partially or completely weakened after HPGR operation.

Figures 4–6 illustrate the breakage functions of each size classes of HPGR feed and product samples. An additional series of tests for the impact breakage of $-19 + 12.7$ mm HPGR feed and HPGR product samples under the same 0.44 kWh/t impact energy level was conducted for reproducibility concern. Each of the points showed $\pm 8.5\%$ variation from the original corresponding ones given in **Figure 5**. These figures show that the weakened product samples tend to generate coarser fragments than those generated by non-weakened HPGR feed class. On the other hand, breakage functions of HPGR feed and product samples become identical at excessive impact energy levels. It is also interesting to note that breakage functions seem to deviate from linearity by giving more or less S-shaped curves mentioned within Section 1.2. This indicates that there were discontinuities (pores and/or cracks) within both feed and product samples altering their fragmentation patterns from linearity. **Table 3** shows that the values of $A*b$ estimated from t10-Eis data (defined in Section 2.1) are higher in HPGR feed than those of HPGR product samples for all the tested size levels. Hence, HPGR feed samples were more amenable to produce finer

fragments than HPGR product samples upon impact. **Table 4** shows the overall averages of micro-hardness results for feed and product classes along with the standard deviation. It is clear that HPGR product was distinctively softer than HPGR feed. On the other hand, the standard deviation found for HPGR feed was higher meaning that hardness among feed particles was fluctuating in a wider range. BET results on **Table 5** show that both total pore volume and single-point surface area values were higher for the feed sample suggesting the fact that the tested feed sample was more porous than the product sample.

Radiographic traces of particles from both classes of HPGR feed and product are given in **Figure 7** and **8**, respectively. It is clear that there was an explicit domination of the darker regions within the brighter bulk matrix for almost all feed particles. This domination by the darker regions was much less severe in product particles as can be seen from **Figure 8**. The results show that HPGR feed particles were severely porous while HPGR product particles were highly intact with much less pore texture.

In **Figure 9**, a collection of secondary electron and software processed SEM images of HPGR feed and product particles were listed. **Table 6** shows the overall averages of mean, standard deviation, maximum and minimum values for sphericity, roundness and mean spherical diameter for both HPGR feed and product particles subjected to SEM and image processing examinations. All of these three parameters were found to have lower values for product class than for feed class. In other words, the surficial discontinuities tended to shrink to smaller diameters (decreased mean spherical diameter) by having sharper edges/corners (decreased roundness) and more irregular shapes (decreased sphericity) after compressive nature of HPGR operation. In **Table 7**, all the aforementioned results obtained for the HPGR feed and product samples are comparatively summarized.

DISCUSSION

The reason of higher breakage probabilities illustrated in **Figure 3** in HPGR product samples can be correlated to the superior effect of micro/macro-cracks within particles by means of crack initiation and propagation. In this case, HPGR greatly reduced the porous structure within clinker and produced a uniform but micro/macro-crack-containing texture (**Table 4** and **Figure 8**). Even the spherical pores that tended to survive should have become elongated by compressing HPGR and acted as cracks or created some kind of stress concentration sites at their sharp edges/tips (**Table 6; Figures 8, 9-c, d, e3 and e4**). All such defects concentrate loaded stresses more readily than more spherical pores found in feed samples, and the uniform almost-pore-free texture in product particles provides easier crack propagation patterns due to less interaction between propagating crack and pores. On the other hand, the situation within feed particles is different because of highly severe and homogeneously distributed network of pores in their matrix (**Table 5, Figures 7, 9-a, b, e1 and e2**). This severe network of pores somehow interacts with the propagating cracks. On comparing the breakage probability values of samples, the feed material is stronger than the product sample which could have been caused by these interactions. However, such networks should also result in finer fragmentation once the particle is broken or failed under impact. Considering that the impact breakage functions of feed and product samples reflect fragments generated after failure, finer fragments generated with HPGR feed sample should be a consequence of the crack propagation through this severe and homogenous pore network (**Table 3, Figures 4–6**). It should also be noted that the above statement applies only for cases where stressing levels are sufficiently low to give a crack-related failure. On the other hand, it is seen from **Figures 4–6** that feed and product samples give especially the same fragments when impact

energy levels are extremely high. In this case, shattering should prevail thereby disintegrate the feed and product particles to the same extent regardless of the nature of their discontinuities.

CONCLUSIONS

HPGR weakens the selected coarse-size clinker. Then, the breakage probabilities of weakened clinker particles will increase in the impact breakage process. These weakened samples produce coarser fragments upon impact loading that leads to coarser cumulative breakage functions than those generated by the samples of HPGR feed (i.e., non-weakened samples) under the same specific impact energy.

The variations in the impact breakage parameters before and after HPGR operation most probably occurred only because of the alterations in the discontinuities within particles. It transformed the clinker particles from a highly porous structure into an intact and much less porous solid texture. Even the surviving pores adopted less spherical and sharper forms that acted as stress-concentration sites thereby reducing the hardness of clinker. This operation also randomly induced micro-cracks in the particles that were detected by radiographic testing. Although these HPGR-induced cracks are efficient to ease the impact-failure of clinker particles, elimination of their severe pore network after HPGR inhibits finer fragmentation under impact. Structural defects do not affect the fragmentation mechanism when it is in shattering form and occurs at excess stressing levels. Overall, for clinker particles whose size is above 9.53 mm, using HPGR as a pre-grinder cannot promote the generation of finer products at subsequent impact stages while it still keeps the advantage of weakening the clinkers.

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Table 1. Specific impact energy levels (kWh/t) for each size levels of HPGR feed and product samples

Size Levels	Specific Impact Energy Levels (kWh/t)
–25.4 + 19.0 mm	0.01, 0.05, 0.11, 0.22, 0.59, 0.88
–19.0 + 12.7 mm	0.03, 0.11, 0.22, 0.44, 0.88
–12.7 + 9.53 mm	0.03, 0.11, 0.22, 0.44, 0.88, 1.32, 1.70

Table 2. Chemical compositions of HPGR feed and product samples with AR, SR and LSF values

Clinker Constituents	HPGR Product (%)			HPGR Feed (%)		
	Single-Size Levels			Single-Size Levels		
	-25.4 + 19 .0 mm	-19.0 + 12 .7 mm	-12.7 + 9. 53 mm	-25.4 + 19 .0 mm	-19.0 + 12 .7 mm	-12.7 + 9. 53 mm
CaO	61.27	57.17	57.28	58.89	61.94	59.12
SiO ₂	17.90	16.99	15.29	17.00	18.80	16.58
Al ₂ O ₃	4.91	4.60	3.97	4.56	5.30	4.17
Fe ₂ O ₃	3.17	3.72	3.06	3.31	3.42	3.35
SO ₃	1.34	1.38	1.28	1.41	1.25	1.32
MgO	2.03	2.06	1.72	1.93	2.27	1.84
K ₂ O	0.60	0.29	0.80	0.63	0.24	0.86
Na ₂ O	0.76	0.58	0.23	0.75	0.61	0.15
LOI	7.63	12.14	15.76	11.01	5.39	11.85
Total	99.61	98.95	99.39	99.47	99.22	99.25
AR	1.55	1.24	1.30	1.38	1.55	1.24
SR	1.06	1.03	1.16	1.07	1.01	1.10
LSF	2.22	2.04	2.17	2.16	2.15	2.20

Table 3. Estimated functional parameters of A, b and their product for each size-level of HPGR feed and product samples

Size Fraction	E_{is} (kWh/t)	HPGR Feed				HPGR Product			
		t10 (%)	A	b	A*b	t10 (%)	A	b	A*b
-25.4 + 19.0 mm	0.88	41.15	63.29	1.20	75.95	35.50	66.44	0.88	58.63
	0.59	32.09				27.80			
	0.22	14.73				11.12			
	0.11	7.76				5.70			
	0.05	3.31				3.12			
	0.01	0.77				0.71			
-19 + 12.7 mm	0.88	38.46	54.83	1.36	74.57	37.38	59.50	1.13	67.24
	0.44	24.17				23.68			
	0.22	14.35				12.95			
	0.11	8.30				6.57			
	0.03	2.35				1.89			
-12.7 + 9.53 mm	1.70	53.29	66.52	1.00	66.52	52.45	71.12	0.80	56.90
	1.32	50.37				47.01			
	0.88	39.43				36.00			
	0.44	23.64				21.78			
	0.22	13.23				10.45			
	0.11	4.87				5.32			
	0.03	1.36				1.47			

Table 4. The mean, standard deviation and coefficient of variation for micro-hardness (HV) values taken from polished sections of HPGR feed and product particles

Particle Class	Size	Statistics	HPGR Feed	HPGR Product
-19.0 + 12.7 mm		Mean	370	285
		Standard Deviation	81	29
		Coefficient of Variation	22	10

Table 5. Single and multi-point BET analyses results for HPGR feed and product samples (P/P₀: Ratio of the applied pressure to the vapor pressure)

Sample Class	Particle Size Class (mm)	Total Pore Volume (cm ³ /g)	at P/P ₀	for Pore Diameter (Å)	Single Point Area (m ² /g)	at P/P ₀
HPGR Feed	-19.0 + 12.7	3.053×10^{-4}	0.96084	< 511.9	0.14	0.31174
HPGR Product	-19.0 + 12.7	2.822×10^{-4}	0.96104	< 514.4	0.03	0.36104

Table 6. Sphericity, roundness and mean spherical diameter of surficial pores of HPGR feed and product samples

Values	–12.7 + 9.53 mm HPGR Feed			–12.7 + 9.53 mm HPGR Product		
	Sphericity	Roundness	Mean Spherical Diameter (μm)	Sphericity	Roundness	Mean Spherical Diameter (μm)
Mean	0.8174	0.5783	1.1103	0.800	0.489	0.851
Standard Deviation	0.0153	0.0369	0.2518	0.029	0.040	0.090
Minimum	0.7760	0.5060	0.7190	0.603	0.406	0.735
Maximum	0.8420	0.6310	1.5000	0.837	0.585	1.210
Total Counts of Pores	149342			76611		

Table 7. Comparison of all results of all analyses.

Analysis	Parameter/Property	HPGR Feed	HPGR Product
XRD	Mineralogical Composition	Same	Same
XRF	Chemical Composition	Same	Same
Breakage Probabilities	Strength	Stronger	Weaker
Impact Breakage Distribution Functions	Fragmentation	Finer	Coarser
t10-Eis Relationship	A*b	Higher	Lower
Micro-hardness	Vickers Hardness (HV)	Harder	Softer
Single and Multi-point BET	Total Pore Volume (cm ³ /g)	More porous	Less porous
	Total Surface Area (m ² /g)	Higher	Lower
Radiography	Outer Zone Defects	Porous and Thick	Porous and Thin
	Central Defects	Porous to Less Porous	Almost Pore-free (some with cracks)
SEM and CLEMEX©	Pore Sphericity	0.8174	0.800
	Pore Roundness	0.5783	0.489
	Pore Mean Diameter (µm)	1.1103	0.851

Figure 1. The size distributions of feed and product samples taken from an industrial, open-circuit HPGR.

Figure 2. XRD traces of HPGR product (top) and HPGR feed (bottom) (Single-size level: $-12.7 + 9.53$ mm).

Figure 3. Breakage probabilities of each size-level of HPGR feed and product as a function of impact energy

Figure 4. Impact breakage functions of HPGR product and HPGR feed (Single-size level: $-25.4 + 19.0$ mm).

Figure 5. Impact breakage functions of HPGR product and HPGR feed (Single-size level: $-19.0 + 12.7$ mm).

Figure 6. Impact breakage functions of HPGR product and HPGR feed (Single-size level: $-12.7 + 9.53$ mm).

Figure 7. Radiographic traces of HPGR feed particles (Single-size level: $-19.0 + 12.7$ mm and $-12.7 + 9.53$ mm).

Figure 8. Radiographic traces of HPGR product particles (Single-size levels: $-19.0 + 12.7$ mm and $-12.7 + 9.53$ mm). Arrows show some indicated macro-crack patterns.

Figure 9. SEM images of HPGR feed (a–b), HPGR product (c–d); processed image of feed (e1–e2) and product (e3–e4).

Research Highlights

- The effect of high pressure grinding rolls (HPGR) on the breakage behavior of coarsely-sized Portland cement clinker was studied by drop weight testing.
- Radiographic testing, Vickers micro-hardness, single and multipoint BET, SEM, XRD, XRF, and image processing methods were applied for bulk and surficial porosity characterization.

- HPGR operation induced micro-cracks, decreased the micro-hardness of clinkers together with sphericity, roundness and mean spherical diameter of the porosity within particles.
- HPGR increased impact breakage probability of the tested clinker particles; however, these resulted in coarser fragments produced upon impact.
- The cracks induced by HPGR have a more pronounced effect on particle failure than the pores have whereas these pores enhanced the fine fragmentation under impact loading.