

High Performance Cements and Advanced Ordinary Portland Cement Manufacturing by HEM-refinement

H. Zoz¹⁻³, D. Jaramillo V.², Z. Tian³, B. Trindade⁴, H. Ren¹, O. Chimal-V⁵ and S. Diaz de la Torre⁵

¹Zoz GmbH, D-57482 Wenden, Germany

²ESIQIE, National Polytechnic Inst., Mexico City, DF 07300, Mexico

³CISRI, Powder Metall. & Envir. Techn., Beijing 100081, P.R. China

⁴FCTUC - University of Coimbra, P-3030 Coimbra, Portugal

⁵Adv. Mater. Res. Center CIMAV S.C., Chihuahua CP 31109, Mexico
info@zoz.de

Abstract

High Energy Milling (HEM) is applied for the grinding of cement and this can lead to substantial refinement ($< 2 \mu\text{m}$) and mechanically activation of the powder particles. The present paper reviews the preliminary studies, explains the novel technique and suggests the route into commercial application. Particular attention is paid to wear results with an applied Si_3N_4 -grinding unit where no substantial wear was found after 4000 h of operation.

Keywords: high energy milling, high performance cement

1. Introduction

Since ancient times, cement has been used as binder material to form concrete structures. Today, Ordinary Portland Cement (OPC) is a super large volume product with thousands of monthly produced tons all over the world. OPC has a particle size distribution (PSD) where 90 % of total particles correspond to $50 \mu\text{m}$, disclosing an onset setting time of 2 to 3 hours. Depending on its chemical composition OPC might attain $320 \text{ kg}^f/\text{cm}^2$ of compressive strength after 28 days curing. The conventional firing temperature is today about 1450°C .

In this paper we suggest to apply High Energy Milling (HEM) as an innovative processing technique for the commercial production of superfine High Performance Portland Cement (HPPC) in short term range. As an almost natural consequence, then in long term range, this technique should be applied also for the manufacturing of OPC at lower energy consumption and then at finer structure as well.

2. Experiments

For the preliminary studies, a laboratory scale high energy mill (Simoloyer CM01-21) was used. The vessel of grinding unit was lined with Si_3N_4 and the rotor is built up by restorable Si_3N_4 -bulk-blades. The wear resistance of the ceramic grinding units was tested by the milling tests. Figure 1 shows the disassembled grinding unit.

In the preliminary studies, 3 main parameters (time, rotor velocity and powder/ball ratio) have been investigated. The processing time has been varied between 30 and 150 minutes at different powder/ball weight ratios from 1:20 to 1:40.



Fig. 1. grinding unit W01-21m-SiN (flange, rotor, vessel & blind-lid)

3. Results

The as-received (initial) and the as milled powders were characterized by laser diffraction (PSD), XRD, BET and optical microscopy.

The results show that the refined cement has a much more homogeneous microstructure and very small particle size (D_{50} is given at about $2 \mu\text{m}$ by PSD) with a few ($<10 \mu\text{m}$) medium sized particles below $20 \mu\text{m}$ size and one large particle in the order of $100 \mu\text{m}$. The BET-surface area was found to be $15 \text{ m}^2/\text{g}$.

In order to compare the strength of the High Performance Portland Cement (HPPC) by HEM-refinement, samples were tested using a universal testing machine 50-C-21H4 and the results were shown in figure 2. The loading rate of the machine was 900 N/s . Samples for the compressive tests were applied after 1, 3, 7 and 28 days.

The compressive strength results of conventional OPC from around $94 \text{ kg}^f/\text{cm}^2$ after the first day to $320 \text{ kg}^f/\text{cm}^2$ after 28 days curing do agree well with typical expected values as reported by the C-109 ASTM standards. The

HPPC material achieves a compressive strength of 550 kg^f/cm² after the first day and of 1018 kg^f/cm² after 28 days.

In comparison, the refined and activated HPPC achieves a very high early strength where already after 1 day the strength is about 70 % higher than the strength of the conventional material after 28 days. After 28 days, the strength of the HPPC is more than 3 times higher than the corresponding value of the conventional material.

Finally we checked the wear of the milling tools which is important to be estimated in early stage since in case of success, extremely huge production numbers must be considered. The results are in so far surprising and impressive at the same time.

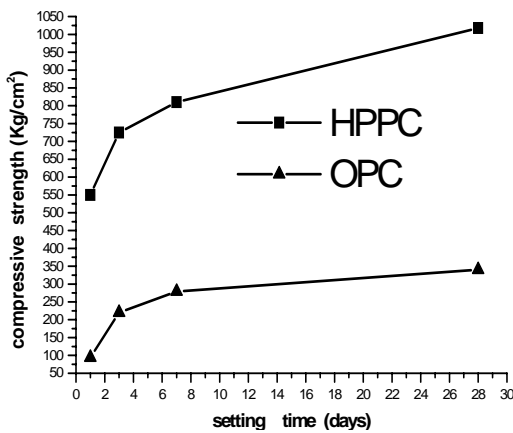


Fig. 2. compressive strength results of HPPC as compared to OPC

Figure 3 shows pictures taken in CIMAV from the disassembled grinding unit after in total 4000 hours of operation with a grinding media load of 2300 g and an OPC-powder load varying between 115-230 g at rotational speeds varying between 500 and 900 rpm. In detail figure 3 shows the inner Si₃N₄-lining of the vessel where in total (all components) 2 cracked plates have been found. One of these cracks is in the area of the given picture and is marked with an arrow. In both cases, no fracture of the plates did fall off which means the cracks are not considered as being problematic, in particular not after that long milling operation at relatively high kinetic.

Figure 4 shows flange and rotor. No cracks or damage could be noticed in the flange-lining and most surprisingly, no damage or cracks could be found at the Si₃N₄-bulk rotor-blades as well. The diameter of the blades has been measured and a diameter-loss of less than 2 mm has been determined. This is a surprisingly very good result and it should be pointed out, that the given total processing time of 4000 hours would refer e.g. to a 24-h-operation for a period of more than 5 months.



Fig. 3. one of two cracks in the lining plates, both appeared in cylinder-lining



Fig. 4. no damage on the rotor-blades, wear surprisingly low, diameter loss less than 2 mm (after 4000 h !)

The YTZ grinding media did not show significant diameter-loss as well.

4. Conclusions (of preliminary studies)

In conclusion, the preliminary studies do show, that HEM/RM is capable to refine and activate OPC which then results into a High Performance Cement with very interesting properties such as high early strength higher than the final strength of conventional material and 3 times higher strengths after 28 days. It illumines a new method to produce much better material at lower cost.

Since the preliminary studies did show a potential, that achievable processing times will be below 30 min and since the experience in the glass-systems did show a cut of processing time of factor 10-50, this seems to open a good chance to achieve final processing times in the range of 1-3 minutes which could then be estimated to a production rate of several tons per day with a single CM100-system. If we then take into account, that Simoloyer-systems are available up to 900 liters size with the potential of further enlargement, then this technique seems to offer the application for the here discussed super large volume product. In any case and from this point of view it will be far capable to produce smaller volumes of High Performance Cement in the range of 10-100 to/day.