Physicochem. Probl. Miner. Process., 55(2), 2019, 575-585

http://www.journalssystem.com/ppmp

Physicochemical Problems of Mineral Processing

ISSN 1643-1049

Received May 12, 2018e; reviewed; accepted August 24, 2018

Comminution behavior and mineral liberation characteristics of low-grade hematite ore in high pressure grinding roll

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Abstract: The comminution process and mechanism of low-grade hematite ore in a high pressure grinding roll (HPGR) were investigated in this work. Three different methods were used for comminution of the hematite ore: HPGR with an air classifier, HPGR with screening followed by ball milling (HPGR+BM), and cone crushing with screening followed by ball milling (CC+BM). In addition, the effects of comminution methods on the degree of liberation of minerals in the grinding products were compared. The results suggest that the comminution process of the ore can be divided into four stages: appearance of tiny cracks, expansion of tiny cracks, which eventually form microcosmic cracks, development of principle cracks, and generation of the conjugate fracturing zone. Intergranular cracks, including partial intergranular cracks and complete intergranular cracks, are apparent in the comminution products of the HPGR. The complete intergranular cracks mainly occur in the conjugate fracturing zone, while the partial intergranular cracks within the particles result in an abundance of locked-particles in the comminution products. Furthermore, the degree of liberation of minerals is more influenced by the final grinding mode rather than the crushing method. It is found that complete intergranular cracks in the conjugate fracturing zone play a significant role in increasing the degree of liberation of iron minerals in the fine size fraction. The “mosaic-type” locked-particles have negligible effect on the liberation properties of iron minerals during the subsequent grinding process.

Keywords: low-grade hematite ore, high pressure grinding roll, comminution behavior, degree of liberation

1. Introduction

With the rapid exhaustion of high-grade iron ore resources, the efficient use of difficult-to-process ores, including hematite and siderite ores, has gained much interest from researchers (Li et al., 2017; Natarajan and Padukone, 2012). There is an abundance of low-grade hematite ores (such as Anshan-type hematite ores) distributed in different regions in China. However, low-grade hematite ores cannot be used directly in blast furnaces because of their low iron minerals content, which are associated with quartz and other gangue minerals (Yu et al., 2017). Therefore, low-grade hematite ores must be treated by mineral processing methods (e.g., flotation and magnetic separation) to obtain the iron concentrate. Prior to separation, the iron minerals must be liberated from the gangue minerals through comminution, including crushing and, if necessary, grinding (Liu et al., 2016; Liu et al., 2014; Yin et al., 2010).

It is crucial to develop an efficient method of reducing the energy consumption of crushing and grinding operations in mineral processing plants, which generally expend more than 50% of the total energy (Han et al., 2012). High pressure grinding roll (HPGR), which has been successfully used to minimize energy consumption in the cement industry since 1985, has now gained popularity for its energy saving potential in various fields (Ni et al., 2011). HPGR offers greater benefits compared with

DOI: 10.5277/ppmp18169

conventional comminution methods, including a 10%-50% reduction in energy consumption, 20%-30% increase in throughput, and significant reduction in operating costs (Benzer et al., 2011; Hilden and Suthers, 2010). These advantages can be attributed to the unique comminution mode of HPGR, especially in the comminution of laminated particle beds (Yuan et al., 2016). The laminated comminution process of HPGR can be divided into three dynamic stages (Lim and Weller, 1999; Han et al., 2012). The materials first enter the space between the rolls under gravity and surface friction of the rolls. In this stage, the compactness of the materials increases from 10% to 45%, and the contact form of the materials transforms from point-to-point contact into surface contact. Parts of the particles are crushed, and surface dissociation takes place. The materials then move into the minimum space between the two rollers (forming laminated particle beds), and the compactness of the materials increases from 45% to ~85%. The interactive forces between the neighboring particles act on all particles under high pressure. Most of the particles are crushed when the pressure strength reaches the crushing strength of the particles. The compressive forces quickly reduce to zero as the materials pass through the minimum space. The compacted particle bed expands due to the release of pressure against it.

In addition to the advantages mentioned above, the results of previous studies suggest that HPGR can enhance mineral liberation considering that particle cracking occurs at the grain boundaries under high-pressures conditions. For example, Celik and Oner (2006) compared the liberation characteristics of clinker minerals ground by HPGR and ball milling, and the results showed that the HPGR produced higher degrees of liberation. Likewise, Ozcan and Benzer (2013) found that the HPGR led to better liberation of copper ores in coarser size fractions, while the degree of liberation of the finer size fractions was almost unaffected by the comminution method. However, other researchers obtained different results. For instance, Solomon et al. (2011) ground a platinum-bearing ore from South Africa by HPGR and ball milling, and they discovered that the ore was not preferentially liberated by the HPGR. Vizcarra et al. (2010) also reported that the liberation behavior of two types of sulfide ores was independent of the breakage method. Based on the results above, the enhanced degree of liberation of an ore by HPGR can be attributed to the mineralogical texture of the ore.

The results of Liu et al. (2017) demonstrated that HPGR reduced the grinding time of low-grade hematite ore compared with conventional operations. In addition, Han et al. (2012) observed that intragranular and intergranular micro-cracks were present in the grinding products of HPGR. Based on these findings, this study was carried out to investigate the comminution process of low-grade hematite ore using HPGR, and to compare the mineral liberation characteristics for different comminution operations.

2. Materials and methods

2.1. Materials

The low-grade hematite ore (particles sizes: 0-12 mm) was collected from the Qidashan Mine in China. The chemical composition and iron mineral phase composition of the ore are presented in Tables 1 and 2, respectively. The assay results show that the ore sample is composed of 27.02 wt.% of total iron (TFe), which mainly exists in the form of hematite and magnetite, 57.64 wt.% of SiO_2, and other components in trace amounts.

Table 1. Chemical composition of the low-grade hematite ore

Constituent	TFe	FeO	SiO_2	Al_2O_3	MgO	K_2O	CuO	P	S
Content (wt.%)	27.02	2.30	57.64	0.54	0.29	0.16	0.37	0.036	0.047

The image of the low-grade hematite ore automatically generated by the mineral liberation analyzer (MLA, FEI Co., Canberra, Australia) is shown in Fig. 1. The results indicate that the ore is primarily comprised of iron minerals (Fe_xO_y) and quartz, and the iron minerals are closely associated with quartz. The composition of iron minerals was further investigated using a scanning electronic microscope (SEM, Hitachi S-3400N, Hitachi Ltd., Tokyo, Japan) integrated with an energy dispersive spectroscope (EDS, Horiba Ltd., Kyoto, Japan). The results show that hematite is the predominant iron mineral and partial hematite forms metasomatic structure with magnetite, as shown in Fig. 2. Figs. 1 and 2 also show that the inherent fractures of the ore sample are minimal, and this will not influence the accuracy of observations of the newly formed cracks during the comminution process. Hereafter, hematite and magnetite are regarded as iron minerals for the sake of uniformity.

Table 2. Iron mineral phase composition of the low-grade hematite ore

Iron phase	Hematite	Magnetite	Siderite	Pyrite	Iron sulfate	TFe
Iron content (wt.%)	15.09	9.18	0.29	0.36	2.10	27.02
Content (%)	55.85	33.98	1.07	1.33	7.77	100.00

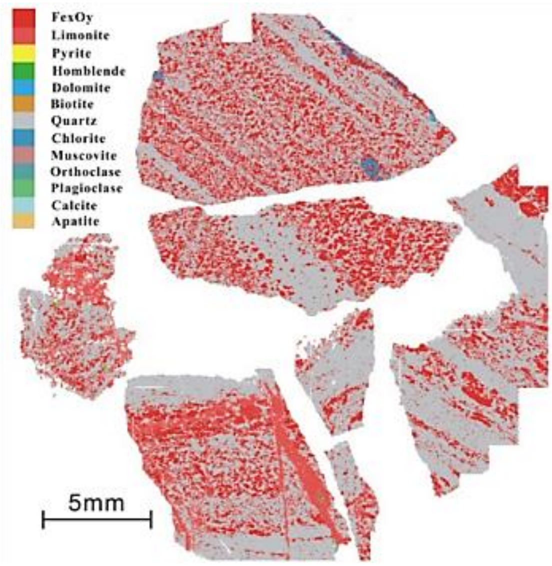

Fig. 1. Image of the ore sample generated by the MLA

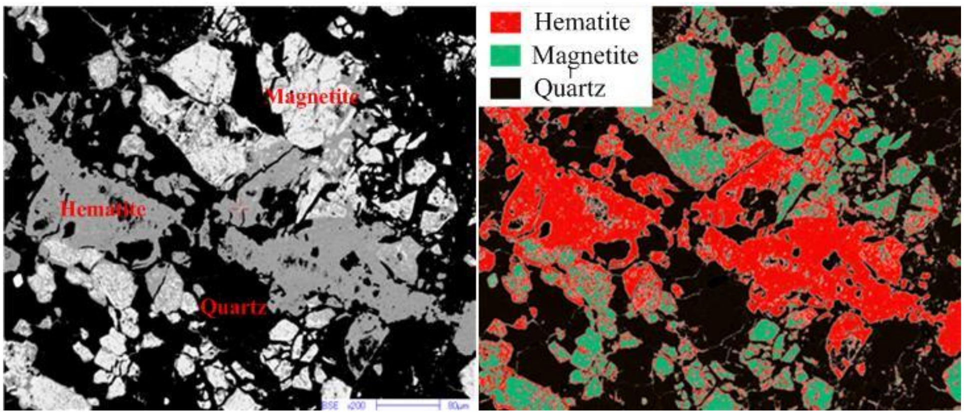

Fig. 2. SEM and line-by-line scanning images of the ore sample

2.2. Experimental procedure

The low-grade hematite ore was ground using a HPGR (CLM-2510, Chengdu Leejun Industrial Co., Ltd, China), which was equipped with two counter-rotating rolls, each with a diameter of 250 mm and width of 100 mm. The work gap between the rolls was set within a range of 4-9 mm (Fig. 3). The results of the comminution of materials using the HPGR are mainly dependent on the peak pressure at the minimum space between the two rollers, which is difficult to measure. Therefore, the specific pressing force, which is the applied grinding force divided by the length and width of the rolls, is typically used to express the working pressure of the HPGR (Hilden and Suthers, 2010). Based on the test results of

Yuan et al. (2016), the comminution tests were performed at pre-determined conditions, with a specific pressing force of 5.2 N/mm^2 and roll speed of 0.18 m/s.

Fig. 3. CLM-2510 HPGR crushing system

The resulting materials collected from the discharge opening of the HPGR were homogenized and separated into ~40 g samples. The samples were mixed with epoxy to maintain the structure of the materials. The polished section of the mixture was produced after drying in a vacuum oven at 353 K. An optical microscope (Leica DM4 P, Leica Microsystems GmbH, Wetzlar, Germany) or SEM was used to examine the expansion of the cracks, particle morphologies of the HPGR products, and the comminution process and mechanism.

To investigate the effects of HPGR on the degree of mineral liberation, the low-grade hematite ore was ground into powder, where 70 wt.% products was passed through a sieve with an aperture size of 0.074 mm. Three different methods were used for comminution of the low-grade hematite ore: HPGR with an air classifier, HPGR with screening followed by ball milling (HPGR+BM), and cone crushing with screening followed by ball milling (CC+BM). The HPGR test was conducted in a closed circuit, where the final product size was controlled by the air classifier. For the HPGR+BM and CC+BM tests, the products from the HPGR and CC were dry screened using a 2 mm screen and the oversized fractions were recycled back to the HPGR or cone crusher. The undersized products were mixed and separated into 500 g samples and then ground to form the final products, where the percentage of solids by weight was 65 wt.%. The degrees of liberation of the final products obtained by the aforementioned methods were measured using the MLA. During the tests, the mineral energy spectra and images of all the samples (each containing 40,000 particles) were generated by the MLA. The degrees of liberation of the minerals were then determined by comparing the results with the standard library of reference spectra, which was achieved automatically using the MLA.

3. Results and discussion

3.1. Comminution process

The expansion cracks and particle morphologies of the HPGR products are shown in Fig. 4. It can be seen from Fig. 4A that tiny cracks are generated in random directions in the ore particles during the initial stage of comminution. These results indicate that the cracks may have mainly occur in weaker regions of the ore, such as in an inherent fracture or on the crystal surface of minerals.

The interactive force of the particles increases with an increase in compactness of the materials. The tiny cracks then increase and gradually become connected to form microcosmic cracks, which are characteristics of conjugate expansion (Fig. 4B). The density of the microcosmic cracks in the HPGR products then increases (Fig. 4C).

Principle cracks form as the number of microcosmic cracks increases, which are also characteristics of conjugate expansion (Fig. 4D). The principle cracks ultimately form the fracture plane of the macro-cracking process.

The conjugate fracturing zone surrounding the new particles is produced under the action of compressive or tensile stresses after the formation of principle cracks (Wills and Napier-Munn, 2006). Some liberated iron minerals, specifically fine-grained iron minerals, are present in the conjugate fracturing zone (Figs. 4E and 4F).

Based on the results, it can be deduced that the single crushing behavior of low-grade hematite ore consists of four sequential stages: appearance of tiny cracks, expansion of tiny cracks, which eventually form microcosmic cracks, development of principle cracks, and generation of the conjugate fracturing zone. However, for comminution of laminated particle beds, these stages may occur in different particles or in different positions of one particle simultaneously.

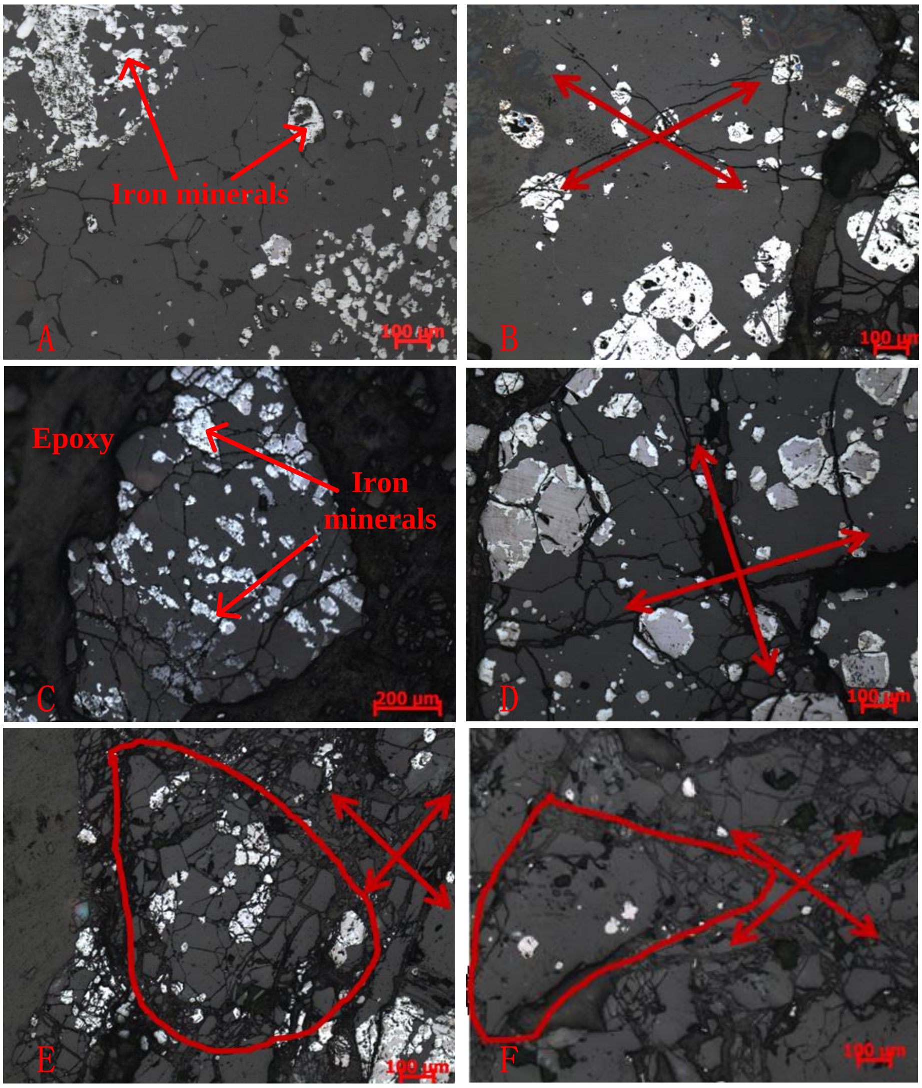

Fig. 4. Microstructures of the low-grade hematite ore products produced by the HPGR

3.2. Intergranular comminution

As mentioned previously, particle cracking at grain boundaries under high-pressure conditions is one of the characteristics of laminated comminution. The intergranular comminution of low-grade hematite ore was further studied and the fracture behavior at the grain boundaries is shown in Fig. 5, while the SEM image and corresponding EDS spectra for a single HPGR particle within the -0.074+0.043 mm size fraction is shown in Fig. 6.

It can be seen from Fig. 5 that some of the fractures occur within the same phase (iron mineral or gangue mineral phase). Beyond this phase, significant quantities of micro-fine iron particles are liberated from the gangue minerals or some parts of their surfaces are exposed, indicating that the partial and complete intergranular cracks are generated in the comminution products of low-grade hematite ore produced by the HPGR. The presence of intergranular cracks is also evident in Fig. 6, in which the main elements of Phases A, B, and C were analyzed by EDS. The main elements of the "a" point in Phase A are Fe and O, indicating that Phase A is the iron mineral phase. The characteristic peaks of Si and O are mainly observed in the spectra of the "b" and "c" points in Phases B and C, respectively, which indicate that Phases B and C are the gangue mineral phases. The obvious crack between Phases A and B is the intergranular crack. The complete intergranular cracks play a major role in improving the degree of mineral liberation in the conjugate fracturing zone. However, there is an abundance of locked-particles present in the grinding products because there are more partial intergranular cracks in the particles.

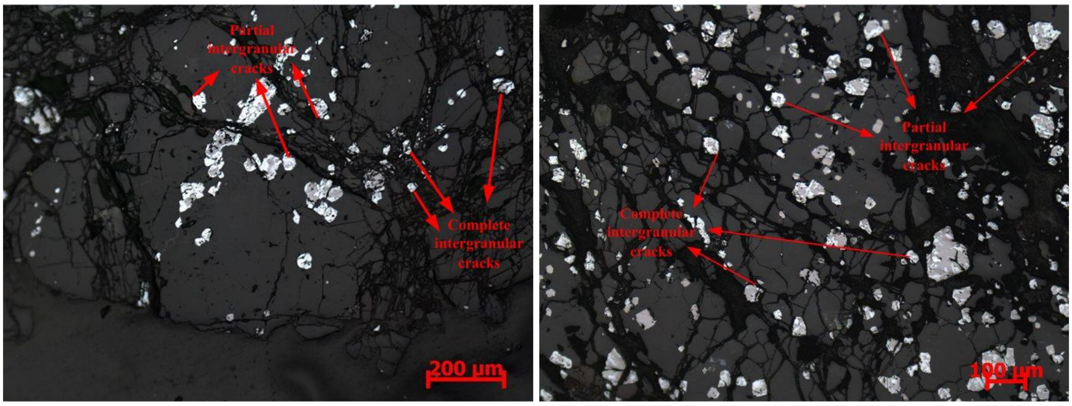

Fig. 5. Fracture behavior of the HPGR products at the grain boundaries

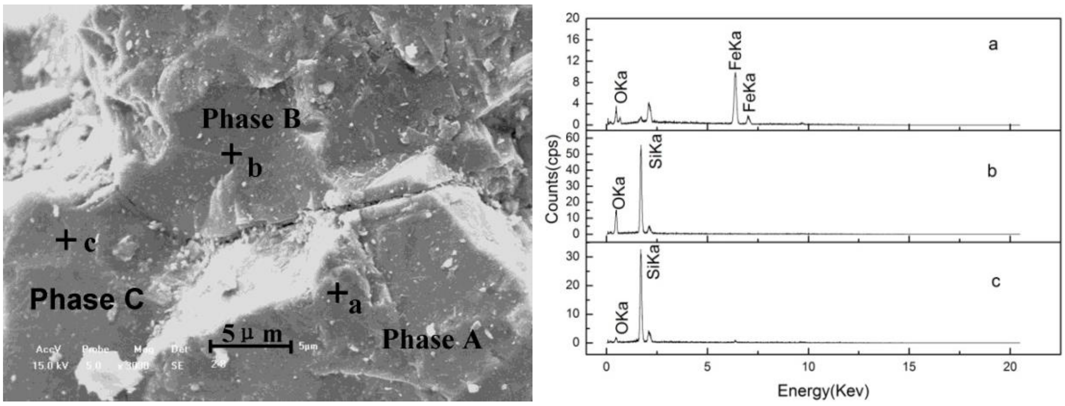

Fig. 6. SEM image and EDS energy spectra of a HPGR particle within the -0.074+0.043 mm size fraction

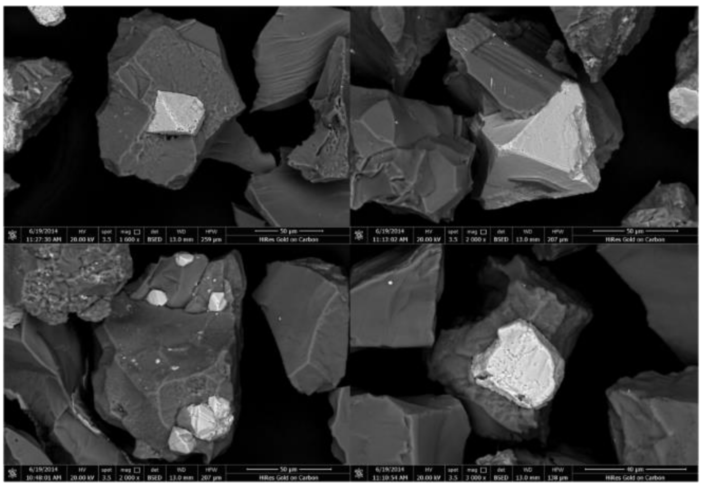

Fig.7. SEM images of the "mosaic-type" locked-particles

The locked-particles were further observed using SEM and the corresponding images are shown in Fig. 7. The results demonstrate that there are obvious partial intergranular cracks in the micro-fine iron particles, as indicated by the grey-white regions. Some areas of the iron particles' surface are exposed, while others are embedded in the quartz. However, the effect of locked-particles on the degree of mineral liberation is not clear from these results; therefore, further investigation is needed.

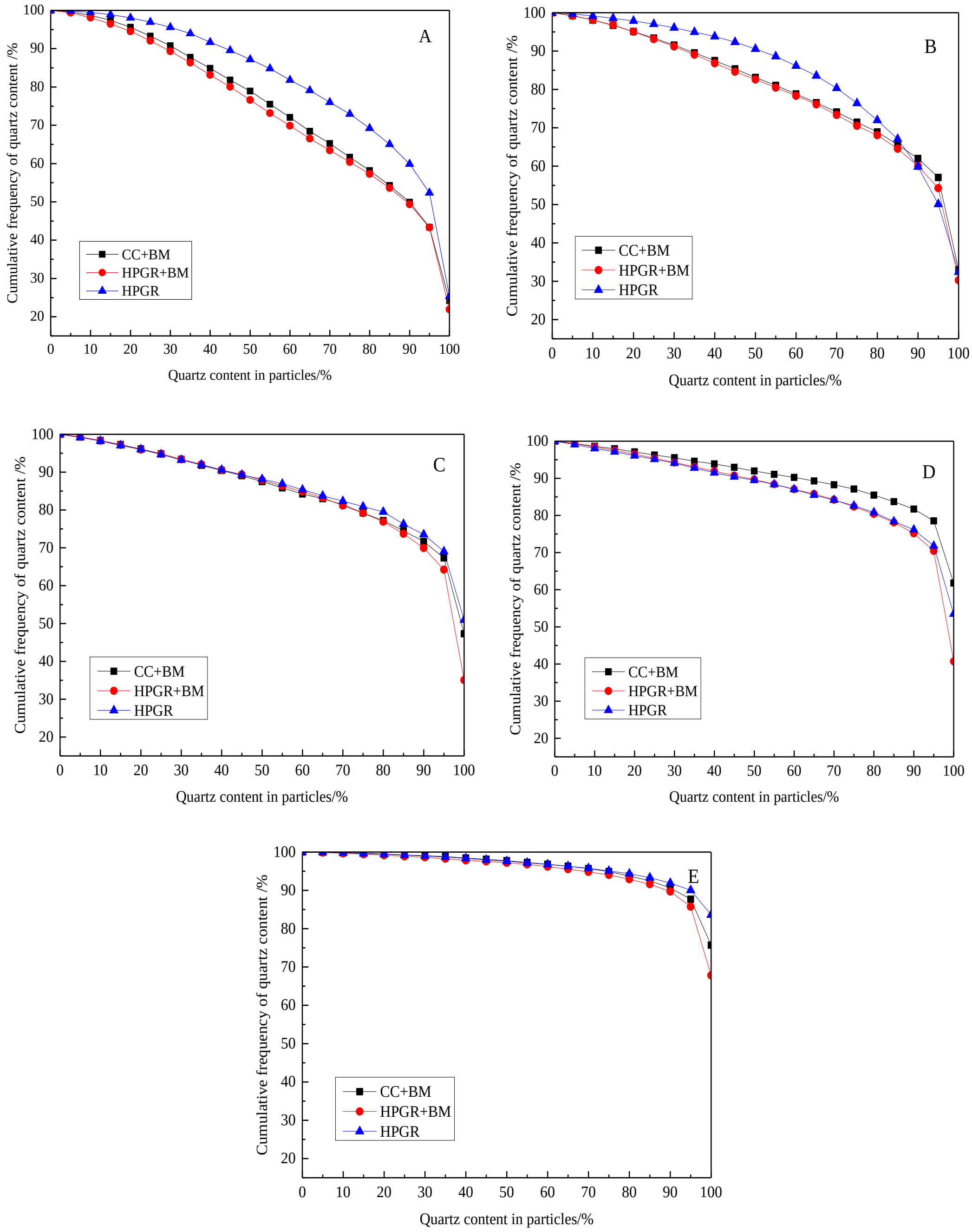

Fig. 8. Degree of liberation curves for quartz in the grinding products: A) +0.100 mm size fraction; B) -0.100+0.074 mm size fraction; C) -0.074+0.043 mm size fraction; D) -0.043+0.031 mm size fraction; E) -0.031+0.015 mm size fraction

3.3. Comparison of the degrees of mineral liberation

The degrees of minerals liberation of the grinding products produced by the HPGR, HPGR+BM, and CC+BM comminution methods were measured using the MLA. In general, the degree of liberation is almost 100% for the -0.015 mm size fraction. The -0.015 mm size fraction is characterized by poor dispersion and ease of adhesion, which will influence the detection accuracy of the MLA. For this reason, the grinding products within the -0.015 mm size fraction were not tested in this work. The degree of liberation curves for quartz and iron minerals in the grinding products are shown in Figs. 8 and 9, respectively.

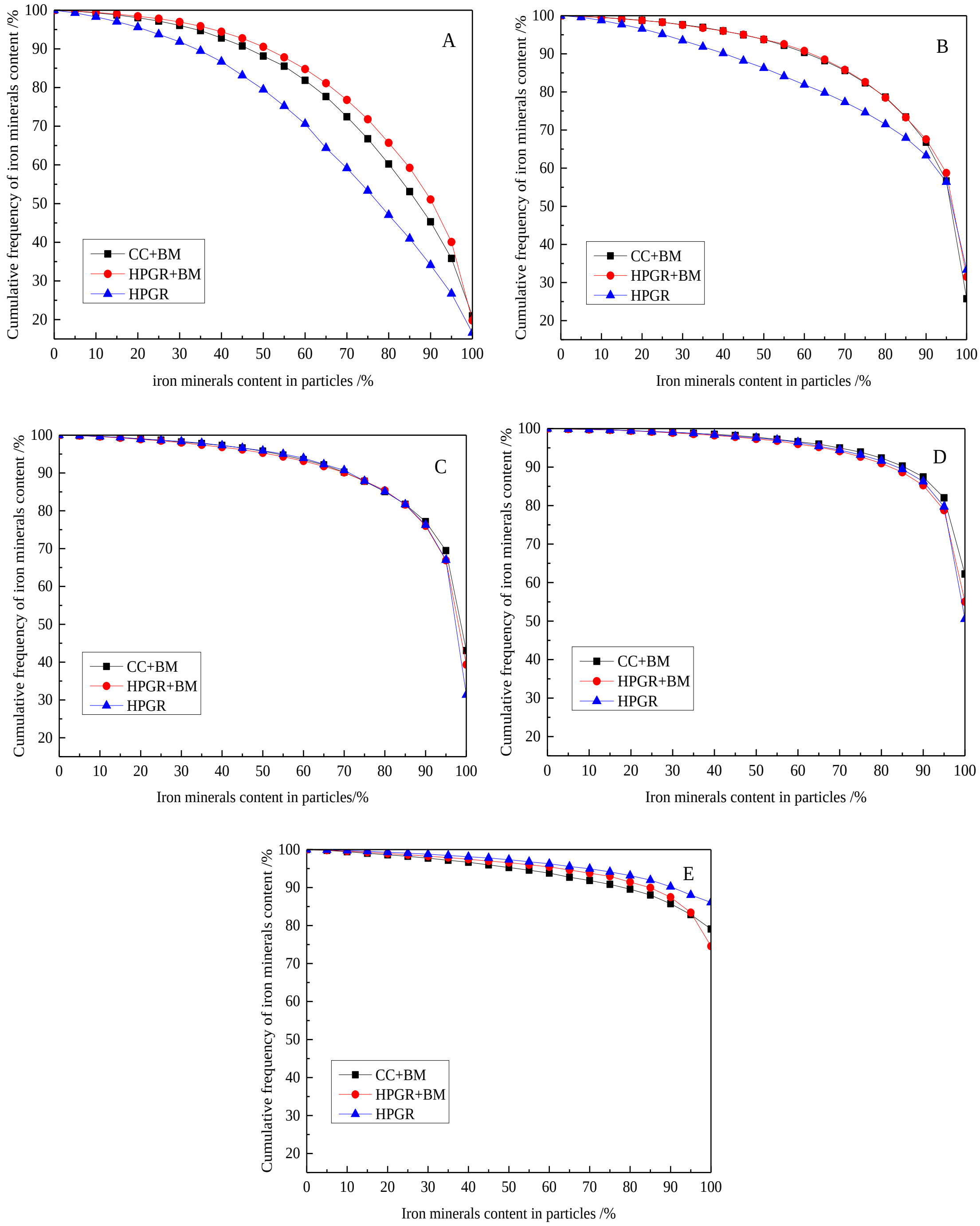

Fig. 9. Degree of liberation curves for iron minerals in the grinding products: A) +0.100 mm size fraction; B) -0.100+0.074 mm size fraction; C) -0.074+0.043 mm size fraction; D) -0.043+0.031 mm size fraction; E) -0.031+0.015 mm size fraction

In general, the iron oxide content of the iron ore concentrate should be more than 90%. Therefore, particles with a single mineral content of more than 90% are regarded as the liberated grains in this study. Figs. 10 and 11 show the percentage of particles in which the liberated quartz and iron minerals are more than 90% based on the results shown in Figs. 8 and 9.

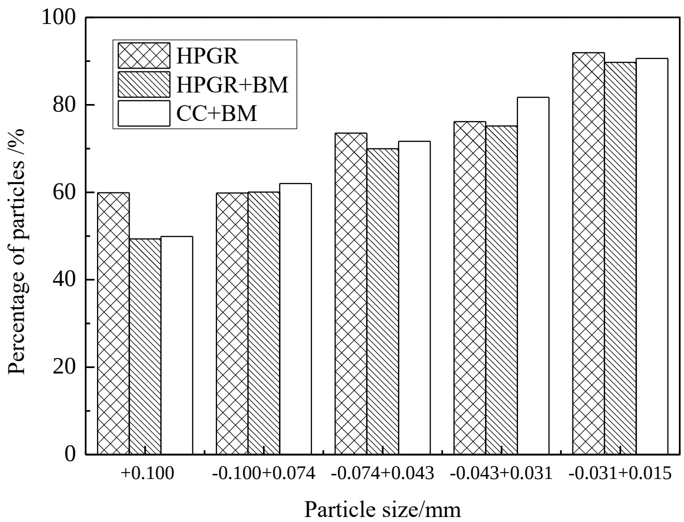

Fig. 10. Percentage of particles in which the liberated quartz is more than 90%

It can be seen from Figs. 8 and 10 that the degree of quartz liberation increases with increasing grain fineness. Overall, the degree of quartz liberation is higher in the HPGR product compared with those in the HPGR+BM and CC+BM products for the +0.100 mm size fraction, with a difference of ~10%. The differences in the degree of quartz liberation for different comminution methods are not obvious for the -0.100 mm size fractions. These results indicate that the HPGR increases the dissociation of quartz in coarse size fractions, which is advantageous for the pretreatment of low-grade hematite ore to remove tailings. Meanwhile, there is no significant difference between the degrees of quartz liberation in the HPGR+BM and CC+BM products. Thus, it can be deduced that the liberation of quartz in low-grade hematite ore is influenced by the final grinding mode rather than the crushing method before grinding.

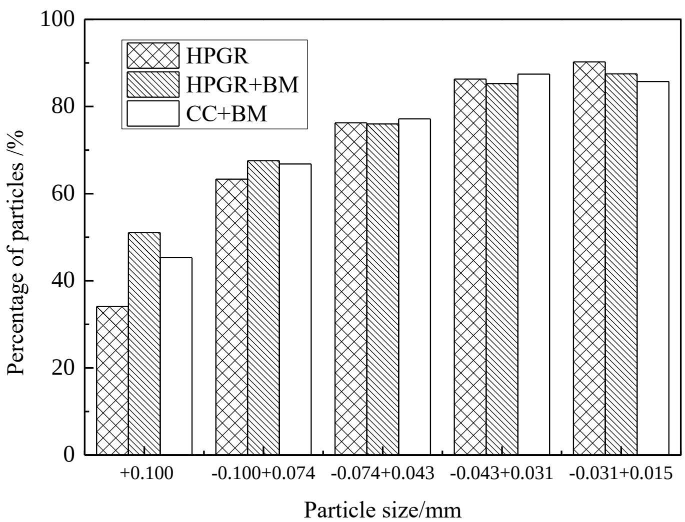

Fig. 11. Percentage of particles in which the liberated iron minerals are more than 90%

It can be observed from Figs. 9 and 11 that the degree of iron minerals liberation also increases with increasing grain fineness. The degrees of iron mineral liberation are higher in the HPGR+BM and CC+BM products compared with that in the HPGR product for the +0.043 mm size fraction. This result emphasizes the significant influence of the final grinding mode on the liberation properties of minerals, including iron minerals. In addition, the degrees of iron minerals liberation in the HPGR and HPGR+BM products increase by 4.49% and 1.73%, respectively, compared with that in the CC+BM product for the 0.031+0.015 mm size fraction. This can be attributed to the presence of intergranular cracks, particularly complete intergranular cracks in the conjugate fracturing zone, as described in Section 3.2. Based on the results, it can be deduced that the "mosaic-type" locked-particles shown in

Fig. 7 have a negligible effect on the liberation properties of iron minerals during ball milling subsequent to the HPGR process.

4. Conclusions

Based on the ore properties, in this work the comminution behavior of low-grade hematite ore in HPGR and the liberation characteristics under different operations were investigated in detail. The following conclusions were drawn based on the findings:

In general, the comminution process of low-grade hematite ore in HPGR can be divided into four stages: appearance of tiny cracks, expansion of tiny cracks, which eventually form microcosmic cracks, development of principle cracks, and generation of the conjugate fracturing zone.

The existence of complete intergranular cracks is apparent in the conjugate fracturing zone of the HOGR products. Meanwhile, numerous micro-fine iron mineral particles exhibit partial intergranular cracks that are partially embedded in the quartz, resulting in the generation of "mosaic-type" locked-particles.

The use of HPGR increases the dissociation of quartz for coarse size fractions. For the +0.100 mm size fraction, the degree of quartz liberation increases by ~10% in the HPGR product compared with those in the HPGR+BM and CC+BM products. There is no significant difference between the degrees of quartz liberation in the HPGR+BM and CC+BM products.

The degrees of iron mineral liberation in the HPGR and HPGR+BM products increase by 4.49% and 1.73%, respectively, compared with that in the CC+BM product for the -0.031+0.015 mm size fraction. This increase can be attributed to the complete intergranular cracks formed in the conjugate fracturing zone of the HPGR product.

Acknowledgments

The authors gratefully acknowledge the National Natural Science Foundation of China (Grant No. 51304175) for funding this research.

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