Investigation of microcrack formation in vanadium-titanium magnetite using different crushing processes

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Synopsis

Characteristics of microcracks in vanadium-titanium magnetite crushed by high pressure grinding roll (HPGR) and conventional jaw crusher (JC) were investigated. In crushing by both HPGR and JC, stress cracks, intragranular cracks, and cleavage cracks were developed. Stress cracking was the initial stage and fundamental basis of fracture. The intragranular cracking could accelerate the comminution process and thus increase the content of fine particles in the crushed products. Cleavage cracking could enhance the liberation of valuable minerals and gangue minerals. Narrow size fraction samples were prepared and characterized by optical microscopy, scanning electron microscopy (SEM), the Brunauer, Emmett, and Teller (BET) method, mineral liberation analyser (MLA), and Bond ball mill. The specific surface area and pore volume of the HPGR products were found to be significantly higher than for JC products due to the presence of abundant microcracks and a higher fraction of fine particles. Compared to the JC products, the HPGR products showed a better degree of liberation and lower Bond ball mill work index (BWI), although the difference gradually decreased with increasing fineness of grind. The application of HPGR not only reduces the energy consumption in the subsequent grinding process, but also optimizes the separation of vanadium-titanium magnetite and improves the TiO_2 recovery in ilmenite.

Keywords

HPGR, microcrack, vanadium-titanium magnetite, crushing process.

Introduction

Comminution (defined as crushing and grinding) accounts for the greatest proportion of the total energy consumed in a processing plant (Abdel-Zaher and Fuerstenau, 2009). Efforts to increase comminution efficiency stem not only from the need for high production rates, but also to reduce the high energy costs associated with the inherent low efficiency of conventional comminution systems and the lower ore grades that are being milled. Schöenert (1988) stated that the most efficient method of particle breakage, in terms of energy utilization, is through compressing the particle bed between two plates, which has been introduced on the industrial scale by the development of the high pressure grinding roll (HPGR).

Comminution in a HPGR is achieved by the compression of a confined bed of particles, thus generating high interparticle stresses, which result in the generation of a greater number of microcracks as well as a higher fraction of fines in comparison to conventional crushing processes (Ghorbani *et al.*, 2013; Aydogan, Ergün, and Benzer, 2006; Torres and Casali, 2009). The HPGR is considered to be a promising new technology, with confined-bed comminution compared to conventional crushing technologies such as jaw crushing which mainly employ stressing between two metal surfaces (Han, 2012; Tavares, 2005).

The HPGR has gained increased attention and popularity, and has been reported to provide numerous metallurgical benefits such as particle weakening, energy saving, increased liberation in cement manufacture, enhanced liberation at coarser sizes for lead-zinc ore, improved cassiterite concentration, the presence of microcracks and higher porosity for sphalerite particles, preferential comminution where induced microfissures follow grain boundaries causing higher reactivity of the product along the microcracks (Tavares, 2005; Celik, 2006; Ghorbani *et al.*, 2011) and preconcentration for low-grade iron ore. The contributions of HPGR technology to enhanced copper column leaching include higher rock matrix fracturing and grain boundary liberation of sulphides (Ghorbani *et al.*, 2013; Chapman, 2013).

Most of the studies have shown that the improvement in liberation with HPGR is due to the generation of microcracks or microfractures, but detailed quantitative information regarding the mechanism, forms of the microcracks, and their influences on the degree of liberation and downstream processes have not been provided yet. Therefore, this study was conducted to investigate the characteristics and influences of microcracks generated in vanadium-titanium magnetite using a HPGR and jaw crusher (JC).

Materials and methodologies

Vanadium-titanium magnetite samples were collected from the Midi concentrator in China. The properties are listed in Table I. The main valuable minerals recovered by the concentrator include ferromagnetic titanium magnetite, weakly magnetic ilmenite, and pyrite. The gangue minerals are mainly titanaugite, feldspar, and chlorite.

A laboratory HPGR was operated at a specific compressive force of 5.6 N/mm² and roll speed of 0.19 m/s. The two counter-rotating rolls are 250 mm in diameter and 100 mm in width, with a static gap operated at 4 mm. A laboratory JC with a feeding port 60 mm in width and 100 mm in length was used to produce a 3–8 mm crushed product. The vanadium-titanium magnetite samples were crushed by the HPGR and JC in closed circuit with a 3.2 mm screen. The microcracks in the different size fractions of the crushed products were characterized using optical microscopy and scanning electron microscopy (SEM), and the specific surface area and pore volume were determined by the Brunauer, Emmett, and Teller (BET) method (Celik, 2006). The Bond ball mill work index test was performed on the –3.2 mm product, and the degree of liberation was studied by the mineral liberation analyser (MLA) (Chapman, 2013). An analysis of the grinding kinetics of the comminuted products was also conducted.

Results and discussion

Influence of crushing process on the size distribution

The effect of the crushing method on the product size distribution is illustrated in Figure 1. For a given feed size distribution and in a closed circuit operation with 3.2 mm screen, the P_{80} of the HPGR products is 1.05 mm while that of the JC products is 1.45 mm.

HPGR products have a more uniform size distribution and contain a greater proportion of fine particles. Compared to a circulating load of 205.56% for the JC circuit, the HPGR has higher efficiency with a lower circulating load of 104.96% because of the confined-bed comminution between particles.

Microcracking of comminuted product

The heterogeneity and discontinuity (fissuring) are basic characteristics for any rock. Particle deformation and fracture are related not only to the heterogeneity but also to the existence of microcracks (Ghorbani *et al.*, 2013).

Comminution by HPGR is a dynamic process which includes an extruding and compacting stage, confined-bed crushing stage, and clustering and expansion stage, corresponding respectively to deformation (Kick's) theory, cracking and extension (Bond's theory), and fracturing and formation of new surfaces (Rittinger's theory). The latter two stages are essential for comminution efficiency and energy saving.

Microcracks in the confined-bed crushing process are first produced and extended in the concentration region of stress cracks and contact point between the surface of the particle and roll surface (Torres and Casali, 2009). With increasing bearing energy between particles, microcracks in the particles continue to grow, converge, and extend until the ore is comminuted. The main mode of breakage in jaw crushing is by impact. Different structural changes would be produced in ore particles as a result of different crushing processes, with different influences on the subsequent grinding and separation processes. Based on the mechanics of rock fracture, this study identified three modes of microcracking, namely stress cracking, intragranular cracking, and cleavage cracking.

Stress cracking

During the process of rock deformation, the mineral crystals with a large elastic modulus and hard texture bear a greater share of the load due to their ability to resist deformation, while the softer crystals have a weaker bearing capacity. Owing to uneven grain size or crystal defects, the softer crystals tend to be in the main stress transmission path. At certain stress levels, the bonding surface of the mineral crystals tends to cause stress concentration and thus crack formation. The stress crack is relatively smooth and narrow (Figure 2), and new fracture surface could be produced under the action of breaking load until the rock is broken by the extension, connection, and growth of microcracks. Stress cracking is the initial stage and the fundamental basis for rock fracture.

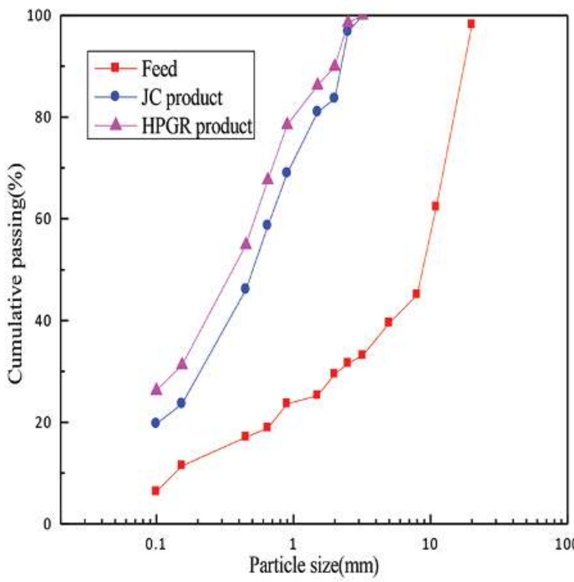

Figure 1—Particle size distribution of vanadium-titanium magnetite crushed by different processes

Table I

Properties of the material

d_{50} (mm)	d_{80} (mm)	H_2O (%)	Mohs hardness	ρ_{bulk} (g/cm³)	Total Fe (%)	TiO_2 (%)	SiO_2 (%)
9.0	15.5	3	4-7	2.56	31.29	11.28	23.64

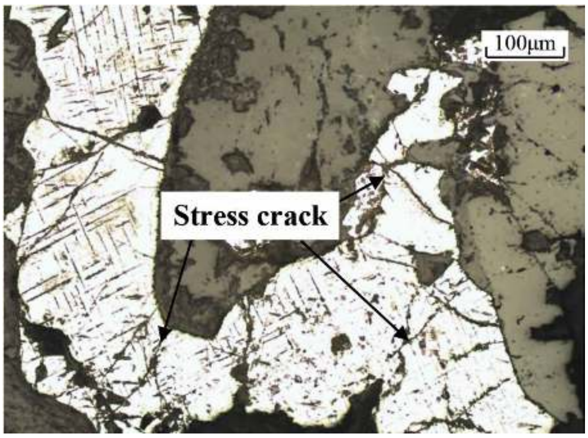

Figure 2—Optical microscopy image of –3.2+2.0 mm size fraction in JC product (40×)

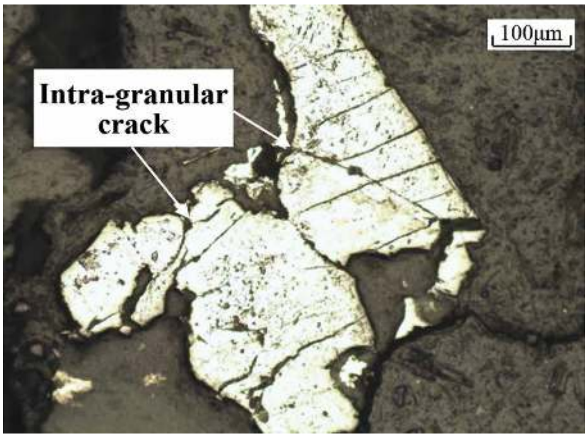

Figure 3—Optical microscopy image of –3.2+2.0 mm size fraction in JC product (40×)

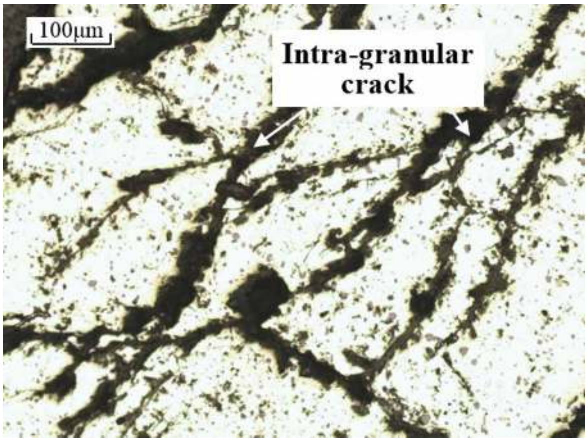

Figure 4—Optical microscopy image of –3.2+2.0mm size fraction in HPGR product (40×)

Intragranular cracking

As the external force and deformation continue to increase, new cracks propagate, especially in the direction of boundaries of individual mineral crystals or the weak structural surfaces due to the crystal defects (King, 1998). Intragranular cracking is one of the most important forms of microcracking in the HPGR products and is mainly produced in the same mineral grain (Figures 3 and 4).

Intragranular cracks in the HPGR products have greater width and depth, concentrated distribution, and also display the phenomenon of bifurcation. As shown in Figures 5 and 6, intragranular cracking were presented that the main elements at points ‘a’, ‘b’, and ‘c’ are Si, Mg, Ca, and O, indicating that phases ‘a’, ‘b’, and ‘c’ consist of similar gangue material.

The intragranular cracks in the HPGR products would accelerate the comminution process of the vanadium-titanium magnetite ore and increase the content of fine particles in the crushed product, which would also improve the specific surface area and reduce the energy consumption in the subsequent grinding process. It is known that intragranular cracking does not influence liberation significantly (King, 1994).

Cleavage cracking

Abundant cleavage cracking is the significant characteristic of confined-bed comminution in HPGR products. Cleavage cracks are generated between adjacent grains of different minerals when the grains of a particular mineral are comparatively loosely bonded in the ore matrix (Figure 7). The cleavage cracks in HPGR products also have greater width and depth. As shown in Figures 8 and 9, the main elements at points ‘a’ and ‘b’ are Fe and Ti, indicating that phase ‘a’ and ‘b’ are similar mineral, but the main elements of ‘c’ are Si, Mg, Al, and O, indicating that phase ‘c’ is a different mineral. The existence of cleavage cracks could not only

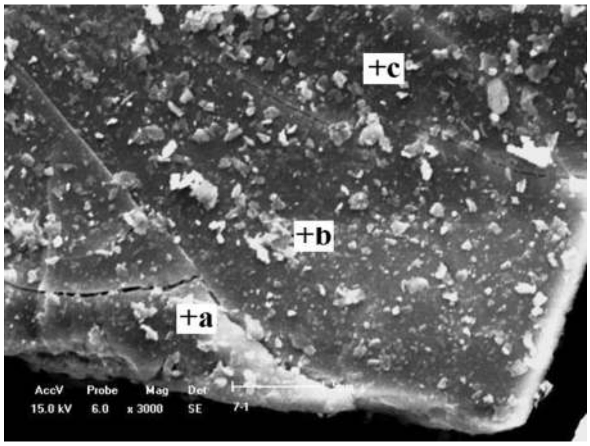

Figure 5—SEM image of an HPGR –0.074+0.045 mm fraction particle

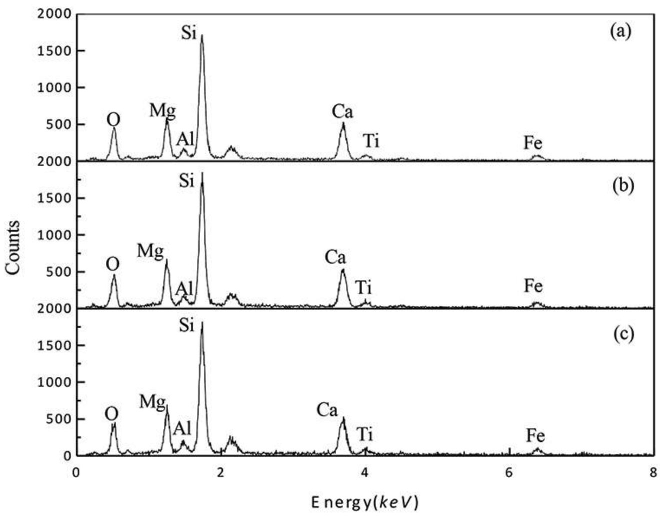

Figure 6—EDS energy spectra of points a, b, and c in Figure 5

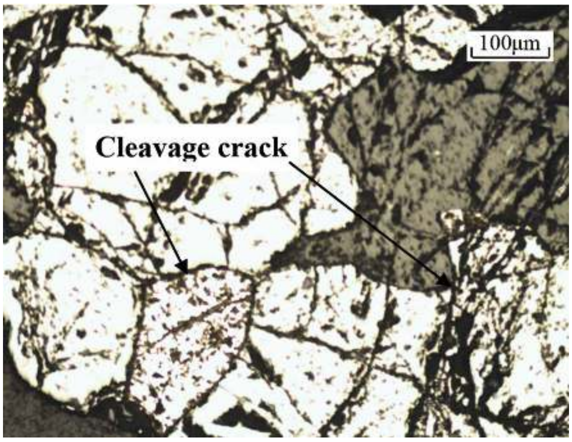

Figure 7—Optical microscopy image of HPGR –3.2+2.0 mm size fraction product (40×)

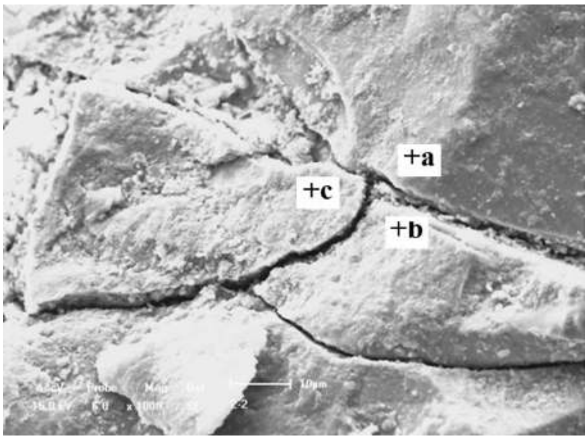

Figure 8—SEM image of an HPGR –2+1mm fraction particle

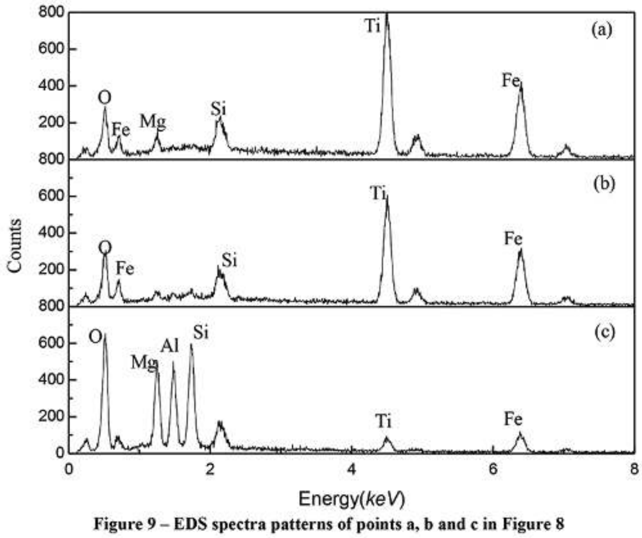

Figure 9—EDS spectra patterns of points a, b, and c in Figure 8

facilitate the reduction of mineral particle size, but also accelerate the separation of different minerals and improve the degree of liberation during the grinding process (King, 1998; King, 1994), which is more favourable for the preconcentration of the low-grade vanadium-titanium magnetite.

Influence of crushing process on specific surface area and pore volume

The microcracks in the crushed product could be considered as pores. The extension and growth of cracks creates new surface area and thus increases the porosity. The specific surface area and pore volume for six size fractions of different crushed products were estimated using the low-temperature N_2 adsorption-desorption method (Han, 2012). Each test sample was 4 g.

Table II shows that the specific surface area and pore volume increase with decreasing product size for both HPGR and JC products. However, the HPGR products have higher specific surface area and pore volume than JC products because of the existence of abundant microcracks and a higher content of fine particles. It is worth noticing that the greatest difference in the specific areas is in the –3.0+2 mm size fraction, which indicates that the HPGR products have much rougher surfaces and more cracks than the JC products in the coarser size fractions. HPGR products have a higher porosity and can be ground more easily than JC products.

Influence of crushing process on Bond ball mill work index

The presence of microcracks in the particles caused by the confined-bed comminution of HPGR could reduce the energy consumption in the downstream grinding process. This reduction was studied through the BWI method (Figure 10).

For a closing screen size of 0.18 mm, the BWI of the HPGR products is 28.80% lower than that of the JC products. With a closing screen size of 0.045 mm, the difference in BWI is 7.81%. It follows from Figure 10 that the ratio of the Bond work indices obtained for jaw crushed and HPGR treated ore increases with an increase in the mesh size of the closing screen used in the Bond test. This observation implies that the number of microcracks per particle decreases with decreasing particle size.

Table II

Specific surface areas and pore volumes of different products

Size fraction (mm)	BET specific surface ($m^2 \cdot g^{-1}$)			DFT pore volume ($\times10^{-3}$ $cm^3 \cdot g^{-1}$)		
	SSJC	SSHPGR	SSHPGR/SSJC	PVJC	PVHPGR	PVHPGR/PVJC
–3.0+2.0	0.109	0.494	4.53	0.6411	0.9002	1.4
–2.0+1.0	0.14	0.612	4.37	1.0942	1.1250	1.03
–1.0+0.45	0.283	0.849	3.00	1.0721	1.3210	1.23
–0.45+0.18	0.314	1.085	3.46	1.1934	1.5620	1.31
–0.18+0.074	0.606	1.373	2.27	1.2211	2.0170	1.65
–0.074	0.737	2.985	4.05	3.9005	5.3800	1.38

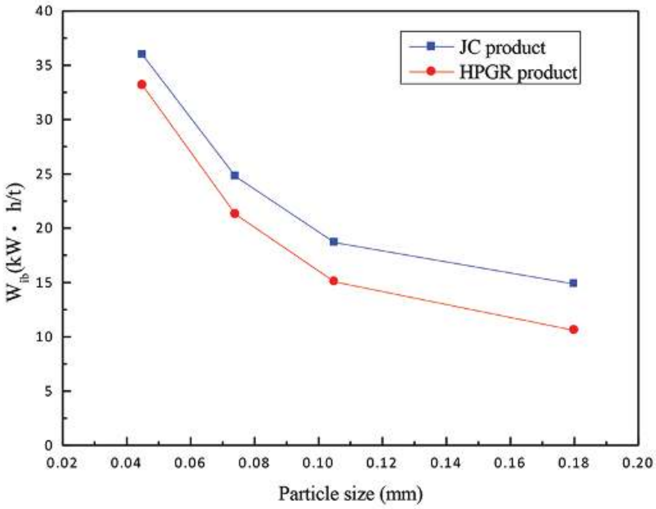

Figure 10—Test results of Bond ball mill work index

Influence of crushing process on liberation at different grinding fineness

The –3.2 mm vanadium-titanium magnetite crushed by different processes was ground to –0.074 mm (undersize respectively accounting for 35%, 55%, and 75%) to test the liberation degree of titanium magnetite by MLA. 'Liberation degree' refers to the percentage of the individual mineral occurring as free particles in the ore in relation to the total content. Statistics and analyses of more than 5000 particles were obtained for each 10 g test sample. The test results are presented in Table III. Individual minerals are mainly include titanium magnetite, ilmenite, pyrite, and titanaugite. Gangue minerals are mainly titanaugite, feldspar, and chlorite.

It can be seen from the results that the liberation degree of HPGR products was higher than that of JC products at a similar grinding fineness. But as the grinding fineness increases, the increasing degree of liberation is gradually reduced. This also suggests that the influence of microcracks produced by HPGR on grinding will gradually decrease with an increase of the grinding fineness.

Grinding kinetics analysis of different crushing products

In order to further investigate the influence of the microcracks generated during comminution on the grinding process, grindability tests were carried out on the –3.2 mm and –3.2+0.074 mm size fractions from the different crushed products (Figure 11). The analysis of the grinding kinetics (Lim, 1996) was conducted using Equation [1]:

$$R = R_0 e^{-kt^n} \quad [1]$$

where R is the percentage of designated coarse fractions in the material after grinding for time t (minutes), R_0 is the percentage of designated coarse fractions in the original material, k is a parameter relating to grinding conditions, and n is a parameter relating to material properties. The greater the value of n, the faster the rate of generation of the designated fine fraction. Taking the natural logarithm of Equation [1] twice, we obtain Equation [2]. The fitting analyses of the grinding kinetic equation according to the grindability test are shown in Table IV.

$$\ln(\ln \frac{R_0}{R}) = n \ln t + \ln k_0 \quad [2]$$

The results showed that the rate of generation of –0.074 mm in ball milling of HPGR products was higher than that of JC

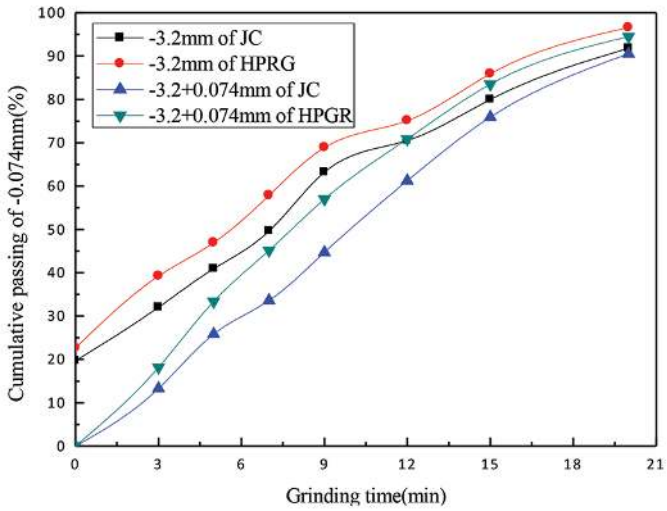

Figure 11—Grindability test results for different crushing products

Table IV

Fitting analysis of the grinding kinetisc

Feed size fraction (mm)	Process	*n*	*k*	R^2
–3.2	Jaw crusher HPGR	1.410 1.478	0.033 0.035	0.994 0.992
–3.2+0.074	Jaw crusher HPGR	2.030 2.066	0.004 0.006	0.996 0.998

Table III

Liberation degree of vanadium-titanium magnetite in different size fractions

% <0.074 mm	Grinding process	Individual minerals (%)	Interlocking mineral (%)		
			Combined with pyrite	Combined with ilmenite	Combined with gangue minerals
35	Jaw crusher] ball mill HPGR-ball mill	83.02 84.94	0.73 0.53	3.02 2.53	13.23 12.01
55	Jaw crusherball mill HPGR-ball mill	88.56 90.72	0.60 0.70	1.99 1.86	8.85 6.73
75	Jaw crusher ball mill HPGR-ball mill	89.98 91.25	0.45 0.60	1.98 1.63	7.60 6.52

products. The generation rate of -3.2+0.074 mm product in ball milling was much higher than that of –3.2 mm product, which once again confirmed that the presence of abundant microcracks in particles could effectively reduce the energy consumption in the grinding process. However, the energy saving gradually reduces with increasing fineness. The microcracks produced by HPGR are more favourable to energy saving during primary grinding of vanadium-titanium magnetite.

Influence of crushing process on separation of vanadium-titanium magnetite

In the conventional separation process for vanadium-titanium magnetite, titanium magnetite concentrate (containing vanadium minerals) with an Fe grade of 55.05% is obtained by stage grinding to 65% passing 0.074 mm and low-intensity magnetic separation. Pyrite by-product and ilmenite are recovered from the tailings after classification and regrinding to 80% passing 0.074 mm. Although the TiO_2 grade of the ilmenite concentrate reaches 47.85%, the complex separation process results in losses of ilmenite in the microfine fraction (–19 µm) during the three-stage grinding.

Parallel separation of titanium magnetite and ilmenite could be realized by low-intensity magnetic separation because of the higher content of fine particles and higher degree of liberation in HPGR products. A strongly magnetic titanium magnetite concentrate with a Fe grade of 54.95% could be obtained with only single-stage grinding (45% passing 0.18 mm) and similar magnetic separation conditions. The weakly magnetic product was reground to 80% passing 0.074 mm and then floated to recover pyrite and ilmenite. The generation of microfine ilmenite was reduced due to changes in the grinding process, which made the particle size distribution of ilmenite more uniform and more amenable to the narrow-scale flotation of ilmenite. The TiO_2 grade of the final ilmenite concentrate was 47.78%, and the TiO_2 recovery increased by 2.66%. The influence of HPGR crushing on the separation of vanadium-titanium magnetite will be described in subsequent papers.

Summary and conclusions

Different tests were performed to evaluate the effect of the comminution process on forms of microcracking in crushed products. HPGR products contain a greater fraction of fine particles than JC products. Stress cracks, intragranular cracks, and cleavage cracks were found in the vanadium-titanium magnetite. The last two types of microscrack increased significantly in HPGR products due to the compression of the confined-bed particles.

Stress cracking is the initial stage and the fundamental basis for rock fracture. The presence of intragranular cracks could accelerate the comminution process and increase the content of fine particles in the crushed product. Cleavage cracks could improve the degree of liberation during the grinding process, which is more favourable for the preconcentration of the low-grade vanadium-titanium magnetite. Microcracks could also improve the specific surface area and pore volume of the HPGR products. The microcracks produced by an HPGR are also more favourable for energy saving in primary grinding, although the energy saving gradually decreases with increasing fineness of grinding.

The grinding and separation process for vanadium-titanium magnetite was changed because of the abundant microcracks and increase content of fine particles in HPGR products. A satisfactory titanium magnetite concentrate could be obtained through only single-stage grinding, and the TiO_2 recovery in ilmenite was also improved.

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