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Microwave assisted grinding of ilmenite ore

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Abstract: The influence of microwave heating on the grinding of Panzhihua ilmenite ore was investigated. Factors that influence the processing are: the microwave exposure time, power density and sample mass. 40 g sample was microwave heated for 30 s with 1 kW of microwave power and followed by water quenching. SEM analysis indicated that intergranular fractures occurred between ores and gangues other than transgranular fractures after microwave treatment, which would liberate minerals from each other effectively. The subsequently magnetic separation trials provided evidence that the recovery rate increased from 44% for raw ore to 72% by microwave treatment.

Key words: microwave; ilmenite ore; magnetite; grinding, magnetic separation

1 Introduction

Accompanied by the depleting of high grade ore reserves, the utilization of low grade ores is increasing in mineral engineering [1−4]. But it is generally believed that the size reduction of ores is an energy-intensive and highly inefficient process. That means more energy is required in future. In the mineral processing, most of the energy is absorbed due to impact and dissipated as heat or noise, while only a small part of energy is used to generate new surfaces [5−7]. Obviously, it is great significance to improve the efficiency of grinding process [8].

During the past century, a large number of researches have been carried out on the grinding processes. Conventional research was concentrated on the improvement in efficiency, and thermally assisted liberation showed a prominent role in improving the efficiency of comminution processing. In some cases, heat treatment would improve mineral liberation by creating intergranular fracture rather than transgranular fracture [9]. Accumulated intergranular fracture would lower the strength of the mineral and increase the grinding efficiency.

However, the economics of conventional thermally assisted liberation maybe limit the industrial application of this technique. WONNACOTT and WILLS [9] presented a research of Cornish tin ore to assess the viability of thermal pretreatment as an economic method of enhancing metallurgical performance. By heating the ore to different temperatures, the cost of heating the ore was from 6.916 to 8.341 dollar/t, and the reduction in required milling power was 0.005 89 dollar/t [10]. If cheaper and more efficient methods of heating could be applied, this technique might become profitable.

In order to improve the economics of thermally assisted liberation, as a great and particular technique, microwave was researched. Microwave is a non-ionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz [11]. When a dielectric material is placed in a microwave field, the dipoles within the material attempt to realign themselves approximately 2.5 billion times per second. This would cause friction inside the material lattice and this friction gives rise to heat [12−13]. Microwave heating is unique and offers a number of advantages over conventional heating, such as rapid heating, material selective heating, non-contact heating.

For over past decades, many researchers have investigated the application of microwave radiation to minerals and extractive metallurgy. 40 minerals were individually researched with microwave heating and divided into two groups: the one that little or no heat was

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generated and the another that considerable heat was generated [14−16]. Generally, most of ores are mixtures of minerals and gangue. The ore can be heated effectively in the microwave field, whereas gangue can not. And the differential heating rates occurring in mineral can induce internal thermal stress. Then fractures generate and the accumulated fractures weaken the mineral, giving rise to the enhancement of the efficiency of thermal treatment and extremely improving the liberation of minerals. A massive sulfide copper ore was investigated under microwave treatment [17−18]. The result showed that a maximum reduction in work index of 70% was achieved after microwave heating for 90 s. Also, microwave heating followed by water quenching was shown to reduce the work index by up to 15% more than for unquenched samples.

In order to make certain the influence of microwave heating upon the mineral comminution, this work investigates the effect of power density, heating time and sample mass upon the grinding of Panzhihua ilmenite ore.

2 Experimental

2.1 Materials

The ilmenite ore used for this study was mined from Panzhihua, Sichuan Province, China. The chemical compositions of the ilmenite ore are listed in Table 1. The analysis revealed that the TFe and TiO_2 are abundant in the ore and the matrix mainly consists of other phases such as SiO_2, CaO, MgO and Al_2O_3. The X-ray diffraction pattern of the raw ore is shown in Fig. 1. According to the XRD analysis, the valuable mineral is mainly presented as magnetite (Fe_3O_4) and ilmenite ($FeTiO_3$).

Table 1 Chemical compositions of ilmenite ore (mass fraction, %)

TFe	TiO_2	SiO_2	CaO	MgO	Al_2O_3	Others
30.67	15.71	20.38	6.48	7.12	3.33	16.28

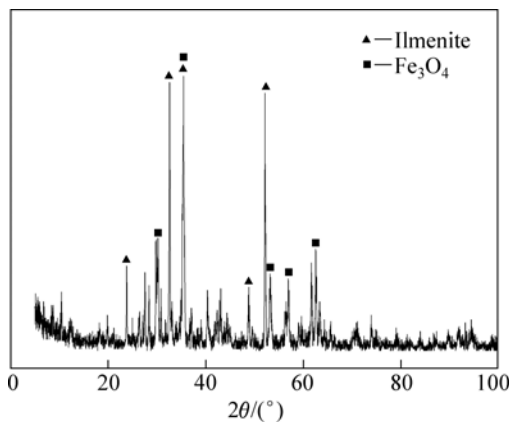

Fig. 1 XRD pattern of raw ore

2.2 Characterization

The products were analyzed by X-ray diffraction to detect the other coexisting mineral phase. XRD pattern was acquired using an X-ray diffractometer (D/Max 2200, Rigaku, Japan) with Cu K_α radiation and a Ni filter operated at 35 kV, 20 mA and a scanning rate of 0.25 (°)/min. SEM (XL30ESEM-TMP, Philips, Holland) was also used to observe the microstructure of the minerals obtained at different experimental conditions.

2.3 Methods

The heating rate test was conducted on representative 40 g ore samples. The microwave used for this experiment is 1 kW of power and 2.45 GHz of frequency. To quantify changes in grindability, the treated and untreated samples were all ground for 30 s by using the sampling crusher. After grinding, the fraction of less than 0.074 mm of the ground specimen was determined by sieve analysis. Samples were heated with time varying between 10 and 60 s. Furthermore, the sample mass in the trials ranged from 20 g to 100 g. Subsequently, magnetic separation trials were carried out to prove the increasing of liberation. In this trial, 40 g representative samples of the untreated and treated material were tested, respectively.

3 Results and discussion

3.1 Mineralogy

The EDAX spectra revealed that the raw ore consists of a certain amount of Fe and Ti (mainly within the light grey part). Subordinate amounts of Al, Mg, Ca and Si also exist (mainly within the dark grey part). Most of the ores inlay in gangues, as shown in Fig. 2.

SEM observation illustrated that the average particle size of mineral grains within the massive ore is approximately between 0.2 mm to 1 mm. So, if the ore was milled to 80% passing sieve with size of 0.074 mm, it could be dissociated effectively, as shown in Fig. 3. It was shown that the mineral grains are disseminated consistently throughout the mineral and the ilmenite and magnetite grains (light grey areas) were inter-grown with larger grains of matrix (dark grey areas).

3.2 Temperature rise curve of ilmenite ore

The heating rate of ilmenite ore by microwave irradiation is shown in Fig. 4. The ilmenite ore could be heated effectively and attained 350 °C in 1 min. Subsequently, the heating rate became slow. The reasons could be attributed to the low power density and sample mass. So in the present experiments, the microwave exposure time was chosen to be 1 min.

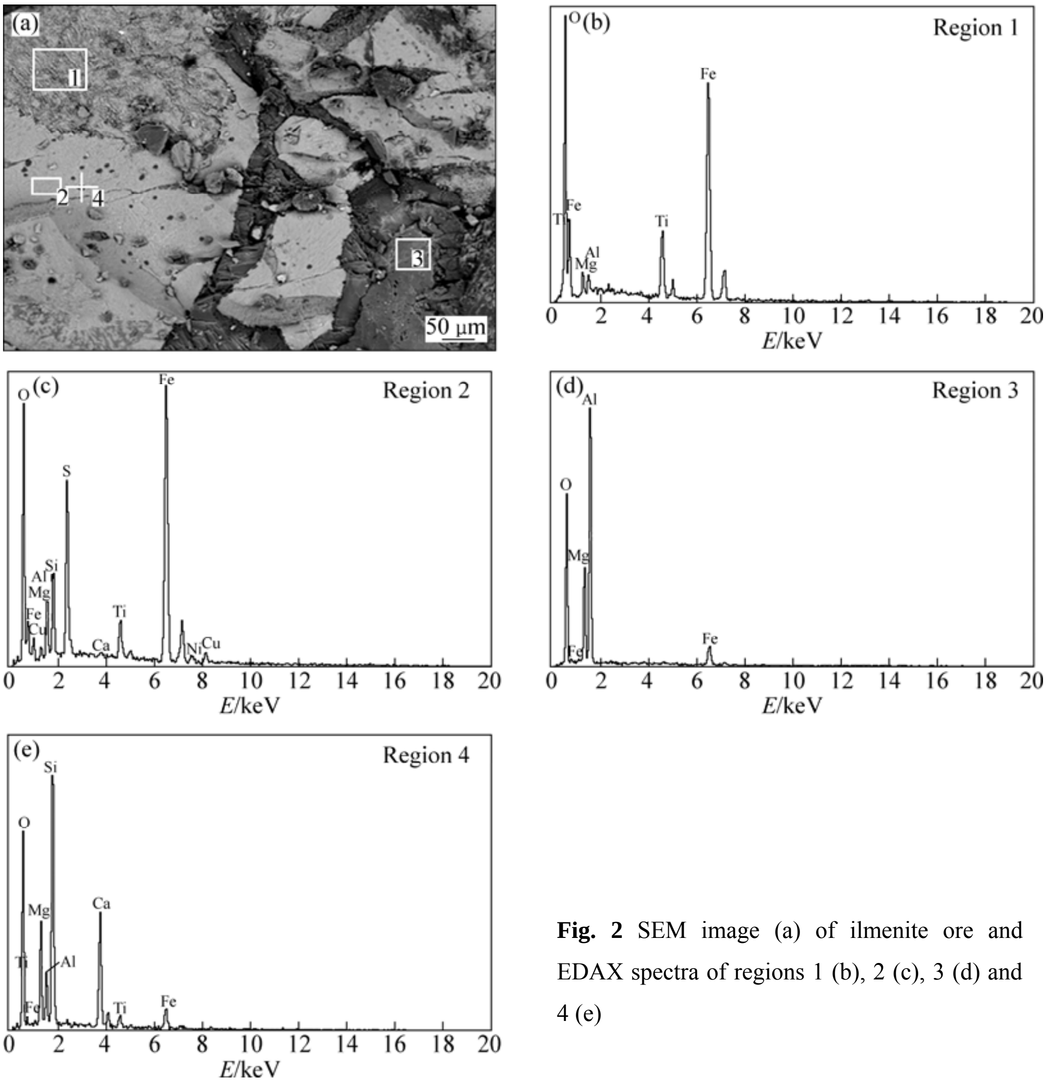

Fig. 2 SEM image (a) of ilmenite ore and EDAX spectra of regions 1 (b), 2 (c), 3 (d) and 4 (e)

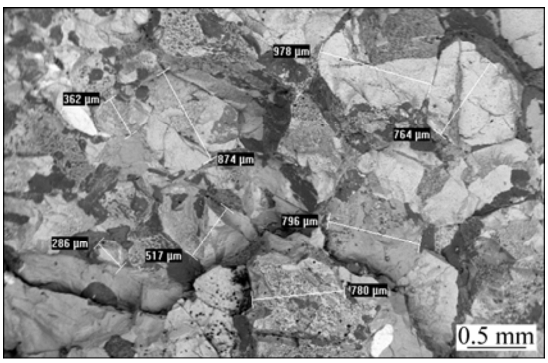

Fig. 3 SEM image showing particle size of ilmenite ore

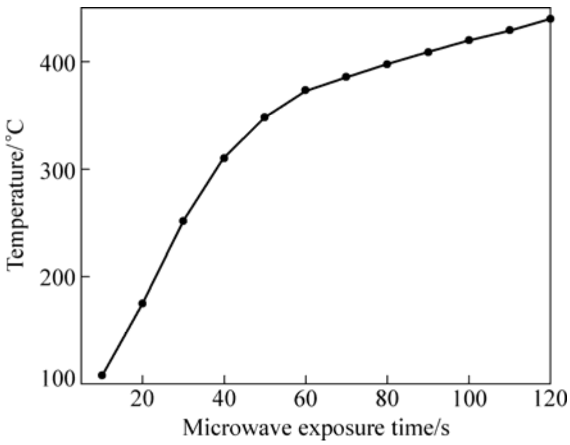

Fig. 4 Temperature rise curve of ilmenite ore by microwave irradiation

3.3 Characterization of ilmenite ore

The XRD pattern of the microwave treated ore is shown in Fig. 5. By comparing with Fig. 1, the peak positions and intensities of magnetite and ilmenite ore were identified distinctly and matched well with the standard XRD pattern. But the peaks of subordinate phases are complicated, mainly including magnesium calcium oxide and other impurity matters. It could be seen from Fig. 5 that there was no phase transformation of the ilmenite ore after microwave heating, or other material generated. However, there were obvious variations in the peak intensities. The apparent alterations in the peak intensities of ilmenite ore and magnetite were identified. The peak intensity of magnetite (Fe_3O_4) and ilmenite ($FeTiO_3$) increased after microwave treatment, and the peak of impurity matters receded or disappeared. The variation in the peak intensities might be attributed to the explanation that more pyroxene monomer was dissociated after microwave radiation, while the polymer of magnetite and ilmenite ore was separated partly.

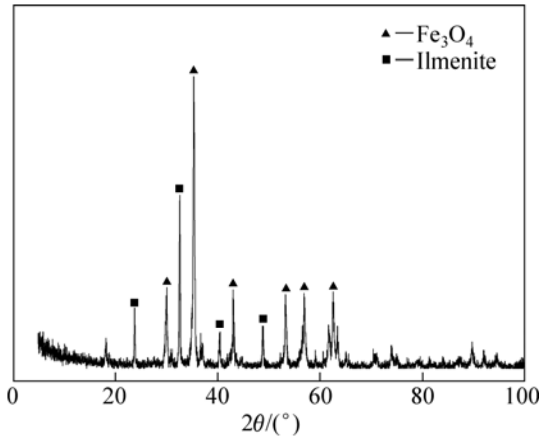

Fig. 5 XRD pattern of microwave treated ore

3.4 Microstructure characterization by SEM

Microstructures of ilmenite ore were characterized by SEM. Figure 6 shows the SEM photograph of ilmenite ore treated with microwave power of 2 kW for 30 s. It is shown that a typical boundary fracture was caused by differential expansion between ore mineral (light grey) and matrix (dark grey). That explained why the fraction of less than 0.074 mm increased after microwave treatment. The fractures caused by microwave treatment made the ore weaker than that of the untreated one [19]. So the ore could be milled easily and the fraction increased.

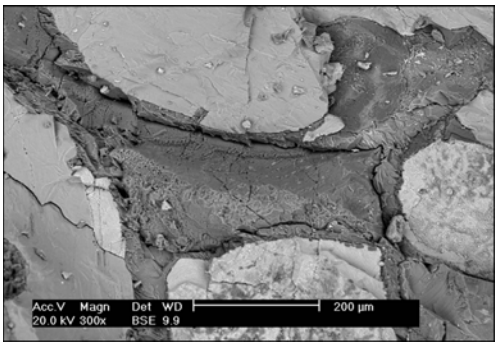

Fig. 6 Typical crystal boundary fracture caused by microwave radiation

3.5 Separation of magnetite

The recovery rates of magnetite at varying microwave power are shown in Fig. 7. It could be seen from Fig. 7 that the microwave radiation had great effects on magnetic separation tests. The microwave treatment had a considerable effect on the recovery of Fe_3O_4. When the ore was microwave treated with 3 kW for 30 s and subsequent water quenched, a recovery rate of 72% was gained, while that of the untreated sample was 44%. And the recovery rates of all treated samples were higher than that of the untreated sample.

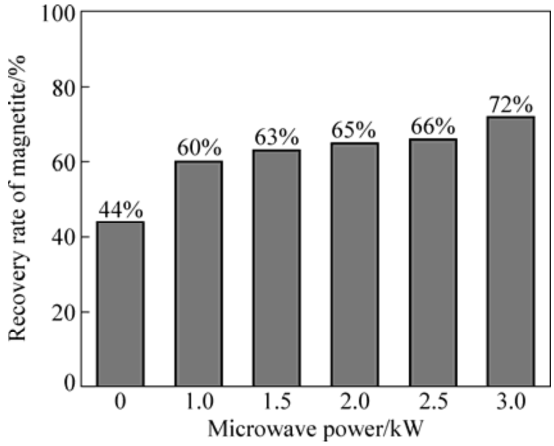

Fig. 7 Recovery rate of magnetite at varying microwave power

This discrepancy could be explained as that the higher microwave power would generate more fractures in the ore than the lower microwave power when the exposure time and sample mass were fixed, then the liberation of the valuable minerals increased. Microwave treatment induces different heating rates in the ore, which leads to different expansion at grain boundaries and the generation of intergranular fractures. As previously stated, intergranular fractures would increase the liberation of mineral, so the recovery rate of magnetite increased. JONES et al [20] and GUO et al [21−22] also concluded that microwave treatment would increase the recovery rate of ilmenite ore.

4 Conclusions

The microwave assisted grinding of ilmenite ore was studied and its structure was characterized using XRD and SEM-EDAX techniques. The results of XRD analysis confirmed that there was no phase transformation of the ilmenite ore after microwave irradiation. The results of SEM analysis indicated that a typical boundary fracture was caused by differential expansion between mineral and matrix. The temperature rise curve indicated that the ilmenite ore could be heated effectively and attained 350 °C in 1 min. The microwave irradiation techniques can be applied effectively and efficiently to grinding processing of ilmenite ore.

References

[1] SCOTT G, BRADSHWA S M, EKSTEEN J J. The effect of microwave pretreatment on the liberation of a copper carbonatite ore after milling [J]. International Journal of Mineral Processing, 2008, 85(4): 121−128.

[2] CHEN G, PENG J H, CHEN J. Optimizing conditions for wet grinding of synthetic rutile using response surface methodology [J]. Minerals & Metallurgical Processing, 2011, 28(1): 44−48.

[3] CHEN J, ZHANG S M, PENG J H, CHEN G, HUANG M Y, LEI Y. Application of the microwave heating technology in the titanium metallurgy [J]. Iron Steel Vanadium Titanium, 2008, 29(3): 68−72. (in Chinese)

[4] ZHAO J W, ZHAO G H, ZHENG Y, XING Z J, MA J R. Application research status of microwave heating in mining and metallurgy [J]. Gold, 2008, 29(12): 39−43. (in Chinese)

[5] JONES D A, KINGMAN S W, WHITTLES D N, LOWNDES I S. Understanding microwave assisted breakage [J]. Minerals Engineering, 2005, 18(7): 659−669.

[6] FITZGIBBON K E, VEASEY T J. Thermally assisted liberation—A review [J]. Minerals Engineering, 1990, 3(1−2): 659−669.

[7] CHEN G, XIONG K, PENG J H, CHEN J. Optimization of combined mechanical activation-roasting parameters of titania slag using response surface methodology [J]. Advanced Powder Technology, 2010, 21(3): 331−335.

[8] CHEN G, CHEN J, PENG J H, SRINIVASAKANNAN C. Dissociation of Ti_2O_3 from titania slag under mechanical activation [J]. Journal of Alloys and Compounds, 2011, 509(24): L244−L247.

[9] WONNACOTT G, WILLS B A. Optimization of thermally assisted liberation of a tin ore with the aid of computer simulation [J]. Minerals Engineering, 1990, 3(1−2): 187−198.

[10] HAQUE K E. Microwave energy for mineral treatment processes—A brief review [J]. International Journal of Mineral Processing, 1999, 57(1): 1−24.

[11] KINGMAN S W, VORSTER W, ROWSON N A. The influence of mineralogy on microwave assisted grinding [J]. Minerals Engineering, 2000, 13(3): 313−327.

[12] KELLY R M, ROWSON N A. Microwave reduction of oxidised ilmenite concentrates [J]. Minerals Engineering, 1995, 8(11): 1427−1438.

[13] XIA H Y, PENG J H, NIU H, HUANG M Y, ZHANG Z Y, ZHANG Z B, HUANG M. Non-isothermal microwave leaching kinetics and absorption characteristics of primary titanium-rich materials [J]. Transactions of Nonferrous Metals Society of China, 2010, 20(4): 721−726.

[14] CHEN G, CHEN J, PENG J H, WANG R D. Green evaluation of microwave-assisted leaching process of high titanium slag on life cycle assessment [J]. Transactions of Nonferrous Metals Society of China, 2010, 20(S1): S198−S204.

[15] ZHANG Z B, ZHANG Z Y, NIU H, PENG J H, ZHANG L B, QU W W, PAN H J. Effects of microwave pretreatment on zinc extraction from spent catalyst saturated with zinc acetate [J]. Transactions of Nonferrous Metals Society of China, 2010, 20(S1): S182−S186.

[16] CHEN G, PENG J H, CHEN J, ZHANG S M. Response surface methodology applied to optimize the experimental conditions for preparing synthetic rutile by microwave irradiation [J]. High Temperature Materials and Processes, 2009, 28(3): 165−174.

[17] VORSTER W, ROWSON N A. The effect of microwave radiation upon the processing of Neves Corvo copper ore [J]. International Journal of Mineral Processing, 2001, 63(1): 29−44.

[18] KINGMAN S W, VORSTER W, ROWSON N A. The effect of microwave radiation on the processing of Palabora copper ore [J]. Journal of the South African Institute of Mining and Metallurgy, 2000, 100(3): 197−204.

[19] WHITTLES D N, KINGMAN S W, REDDISH D J. Application of numerical modelling for prediction of the influence of power density on microwave-assisted breakage [J]. International Journal of Mineral Processing, 2003, 68(1−4): 71−91.

[20] JONES D A, KINGMAN S W, WHITTLES D N, LOWNDES I S. The influence of microwave energy delivery method on strength reduction in ore samples [J]. Chemical Engineering and Processing, 2007, 46(4): 291−299.

[21] GUO S H, LI W, PENG J H, NIU H, HUANG M Y, ZHANG L B, ZHANG S M, HUANG M. Microwave-absorbing characteristics of mixtures of different carbonaceous reducing agents and oxidized ilmenite [J]. International Journal of Mineral Processing, 2009, 93(3−4): 289−293.

[22] WANG Y M, YUAN Z F, GUO Z C, TAN Q Q, LI Z Y, JIANG W Z. Reduction mechanism of natural ilmenite with graphite [J]. Transactions of Nonferrous Metals Society of China, 2008, 18(4): 962−968.

微波辅助磨碎钛铁矿

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摘　要：研究微波加热对攀枝花钛铁矿磨矿性能的影响。微波辐射时间、微波功率密度和样品质量是磨矿过程的重要参数。将 40 g 样品在微波功率 1 kW 下辐射 30 s，然后进行水淬处理。SEM 分析结果表明：经过微波辐射处理的钛铁矿在有用矿物和脉石间产生了裂纹，能够有效地促进矿物与脉石的解离。在随后的磁选实验中，经过微波辐射处理的钛铁矿的回收率从 44%提高到 72%。

关键词：微波；钛铁矿；磁铁矿；磨矿；磁选分离

(Edited by YUAN Sai-qian)