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Enhancement of ilmenite magnetic properties by oxidation roasting and magnetic separation

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Abstract: Oxidation roasting was proposed to treat the problem of the weak magnetic properties of ilmenite. The effects of roasting temperature, roasting time and cooling method on the results of ilmenite magnetic separation were studied. At the same time, the mechanism of the oxidation roasting process was analyzed by XRD, VSM, SEM-EDS. Determine ilmenite suitable oxidation roasting conditions for roasting temperature 750 °C, roasting time of 20 min, and cooling method of air cooling; at this time, the yield of 96.79% can be achieved in the magnetic field intensity of 238.74 kA/m. The XRD results showed that phase changes in the roasting process occur in the order of ilmenite to hematite and rutile to pseudobrookite. The results of VSM showed that the maximum specific magnetization coefficient of ilmenite increased from 3.25×10^{-5} m^3/kg to 5.58×10^{-5} m^3/kg after roasting, and the magnetic property was enhanced. The results of SEM-EDS showed that the phase transition of ilmenite during the roasting process occurs in a spatial order from the edge of the particles to the interior.

Keywords: ilmenite, oxidation roasting, magnetic separation, phase transformation, magnetic properties transformation

1. Introduction

Due to its excellent properties, including low density, high mechanical strength, good plasticity, and high-temperature resistance (Wang et al., 2008; Wei and Niansuo, 2011), titanium is widely used in many fields such as medical devices (Ning and Zhou, 2002), aerospace (Jin et al., 2015), marine industry (Chen et al., 2015), military (Chen et al., 2020), new energy sources (Wu, 2024), communication equipment (Peng et al., 2024) and so on. Titanium minerals are diverse, but the main types currently utilized on the industrial scale are ilmenite and rutile. According to the U.S. Geological Survey (USGS) data (USGS, 2024), as of 2023, the global titanium mineral resources reserve total about 745 million tons (in terms of TiO_2), ilmenite and rutile global resource reserves of 690 million tons, 55 million tons, respectively, accounting for 92.62, and 7.38%. Among them, China's ilmenite reserves are 210 million tons, accounting for 30.43% of the global ilmenite reserves and 28.19% of the global titanium ore resource reserves. Although China is rich in ilmenite resources, most are multi-metallic ores with low titanium grades, having a low comprehensive utilization rate of resources and great difficulty in promoting the grade (Xian and Jian, 2006; Zhang et al., 2016; Chen et al., 2018). The primary ilmenite in China consists of titanomagnetite, magnetite, ilmenite, and silicate gangue. Titanomagnetite and magnetite have strong magnetic properties, while ilmenite and some silicate gangue have weak magnetic properties. Meanwhile, some silicate gangue has non-magnetic properties (Chen et al., 2023). Magnetic separation can effectively separate strong magnetic minerals and other minerals. However, some silicate minerals and ilmenite have similar magnetic properties (Shen, 2021), and obtaining good separation indexes by magnetic separation is difficult. Therefore, in practice, magnetic separation is mainly used as roughing to reduce the subsequent processing capacity. Researchers have carried out extensive and detailed research work to separate titanaugite from ilmenite and thus improved the separation index of ilmenite magnetic separation. Zeng et al. (Zeng et al., 2017) introduced centrifugal force in the separation of ilmenite using a high-gradient magnetic separator. They found that the selectivity of the equipment was enhanced, but when the centrifugal force exceeded a specific value, the

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recovery dropped rapidly. Yuan et al. (Yuan et al., 2021) used superconducting magnetic separation to recover ilmenite. However, in general, the magnetic separation concentrate obtained by the above two methods remains an unqualified ilmenite concentrate product, which must be further separated by flotation.

Ilmenite roasting has been studied by researchers before, mainly including reduction roasting and oxidation roasting. During reduction roasting, Fe in ilmenite is reduced to metallic Fe and Ti is reduced to a series of low-valent compounds (Ti_3O_5, Ti_2O_3, TiC_xO_y, etc.) in a high-temperature reducing atmosphere (Gou et al., 2015). Subsequent weak magnetic separation achieves the separation of Fe from titanium. In oxidation roasting, Fe^{2+} in ilmenite is oxidized to Fe^{3+} in an oxidizing atmosphere at high temperatures. Compared with oxidation roasting, reduction roasting requires a higher temperature and longer roasting time (Chonghui et al., 2023) and consumes a large amount of additional reductant, which results in a much higher energy consumption and production cost than oxidation roasting. In addition, the reduction process is weakly selective and highly susceptible to side reactions (reduction of impurity elements such as Mg, Mn, and Si (Wang and Yuan, 2006) as well as metallic iron catalyzing the oxidation-reduction of CO itself (Dai et al., 1996)). It is impossible to achieve the directional reduction of the specified valuable minerals, which directly leads to decreased product purity, and the control of the test process is complicated. It is highly susceptible to over-reduction (Cheng et al., 2022), which deteriorates the subsequent magnetic separation. Therefore, the industrial use of ilmenite reduction roasting has little value. The previous studies on the oxidation roasting of ilmenite (Bhogeswara Rao and Rigaud, 1975; Gupta et al., 1989; Chen, 1997;1998; Wei et al., 2013; Samanta et al., 2014) have mainly focused on the roasting behavior itself (roasting thermodynamics, roasting kinetics, oxidation mechanism, physical phase transformation, micro-morphological transformation, etc.), while few studies have investigated the effect of oxidation roasting on the subsequent separation of ilmenite.

In this paper, to address the problem of weak magnetism of ilmenite, oxidation roasting was carried out to treat ilmenite to enhance the magnetic properties of ilmenite, and the related mechanism of ilmenite's magnetic transformation was further explored by X-ray diffraction (XRD), vibrating sample magnetometer (VSM), scanning electron microscope - energy dispersive spectrometer (SEM-EDS), which provides a new idea and technical support for the efficient and clean utilization of ilmenite resources.

2. Materials and methods

2.1. Materials

The ilmenite pure minerals were obtained from the strong magnetic separation concentrate from Titanium Industry Co. of Pan gang Co. LTD through multiple magnetic separations and gravity separations in the shaking table. The results of the chemical multi-element analysis are shown in Table 1, which indicate that the TiO_2 grade of ilmenite pure minerals produced is 50.36%, and the purity is more than 95%. XRD results are shown in Fig. 1, which indicates that the prepared samples contain only ilmenite, with few other impurity minerals.

Table 1. Chemical composition of purified ilmenite sample (wt.%)

TiO_2	TFe	FeO	CaO	SiO_2	Al_2O_3	MgO	Mn	S
50.36	26.78	20.26	0.087	1.06	0.38	2.16	0.56	0.049

2.2. Oxidation roasting - magnetic separation

After the muffle furnace (SX2-5-12A) was heated to the set temperature, 10 g of sample was placed in the ceramic crucible, and the time was started. Then, the roasted products were cooled at the end of the scheduled reaction time. The high gradient magnetic separator (L-4-20) was used to perform magnetic separation. The magnetic and non-magnetic products collected were filtered and dried. The magnetic fraction recovery (hereafter referred to as recovery) was calculated based on the solid mass distributions between the magnetic and non-magnetic products.

2.3. Determination of the physical composition of the roasted samples

The physical composition of the roasted samples was analyzed by the X-ray diffractometer (PW3040). Samples ground down to -10μm or less were processed by X-ray diffraction to determine information about the crystal structure. The crystal diffraction patterns were compared to identify the composition of the material phases.

2.4. Determination of the magnetic properties of the roasted samples

The magnetic properties of the samples were detected by the vibrating sample magnetometer (VSM) (JDAW-2000D). Under the control of the computer software IDEAS-VSM Version 4, samples were inserted into the preheated and calibrated device, and the intensity-specific magnetization curves of the samples were measured.

2.5. Observation of microscopic morphology of the roasted samples

The microstructures of the samples were analyzed by the scanning electron microscope (EVO 18 Research), equipped with an EDS (QUANTAX200). The samples were attached to the sample stage using conductive adhesive and observed under vacuum for magnification. The EDS specifications were energy resolution 14 eV, micro-area analysis < 1 μm.

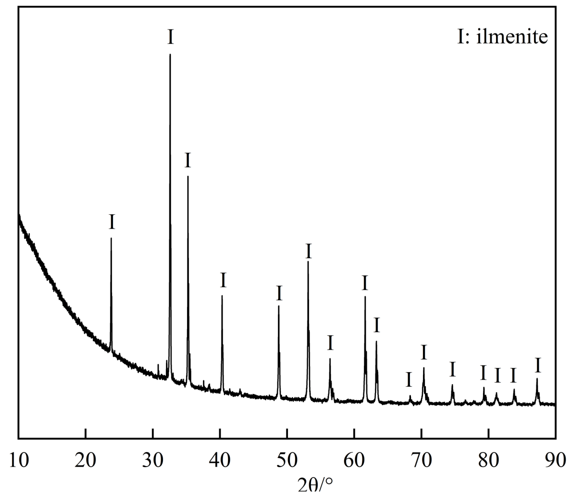

Fig. 1. XRD pattern of ilmenite sample

3. Results and discussion

3.1. Roasting condition test

The effect of roasting conditions on oxidation roasting is significant, and only by optimizing the roasting conditions can an efficient, economical and environmentally friendly roasting process be achieved. According to previous studies on the roasting-magnetic separation of other minerals (Zhu et al., 2002; Song et al., 2007; Yang et al., 2013; Zhu et al., 2019; Ma et al., 2021; Zhu and Qin, 2021; Lei et al., 2022; Luo et al., 2023; Wang et al., 2023a; Wang et al., 2023b), the conditions that can affect the magnetic separation results of the roasting products are mainly the roasting temperature, the roasting time, the cooling method and the magnetic field intensity. Therefore, the effects of the four roasting conditions on the magnetic separation behavior of ilmenite were investigated separately.

3.1.1. Effect of roasting temperature on the results of ilmenite magnetic separation

The roasting temperature affects the oxidation roasting remarkably. The oxidation reaction rate can be accelerated by increasing the roasting temperature. However, a roasting temperature that is too high may lead to over-oxidation or side reactions, deteriorating the roasting effect. Under the condition of roasting time of 45 min, cooling method of air cooling, and magnetic field intensity of 238.74 kA/m, the effect of different roasting temperatures on the behavior of ilmenite oxidation roasting-magnetic separation was investigated, and the experimental results are shown in Fig. 2.

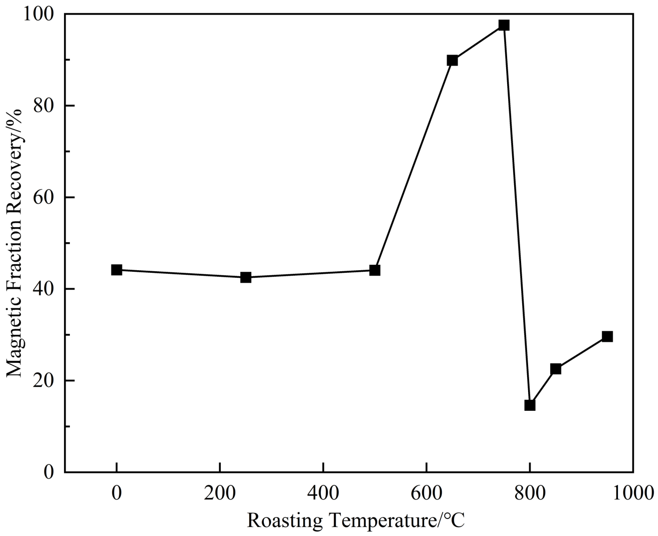

Fig. 2. Effect of roasting temperature on recoveries of ilmenite

As can be seen from Fig. 2, when the roasting temperature is increased from 500 to 750 °C, recovery rises from 44.09 to 96.58%; when the temperature continues to grow to 800 °C, recovery decreases to 14.64%; and when the temperature continues to increase to 950 °C, recovery rises to 29.63%. This is mainly because the high temperature may make part of the ilmenite surface melt to generate strong magnetic metal iron (Mehdilo et al., 2014) and enhance the ilmenite's magnetic property slightly (Cui et al., 2002). Considering the concentrate index and economic cost, the suitable roasting temperature is 750 °C.

3.1.2. Effect of roasting time on the results of ilmenite magnetic separation

Roasting time is also one of the critical factors affecting oxidation roasting. Too short of a roasting time will lead to incomplete oxidation of minerals, while too long will lead to energy waste. Under the condition of roasting temperature of 750 °C, cooling method of air cooling, and magnetic field intensity of 238.74 kA/m, the effect of different roasting times on the index of ilmenite oxidation roasting-magnetic separation was investigated, and the test results are shown in Fig. 3.

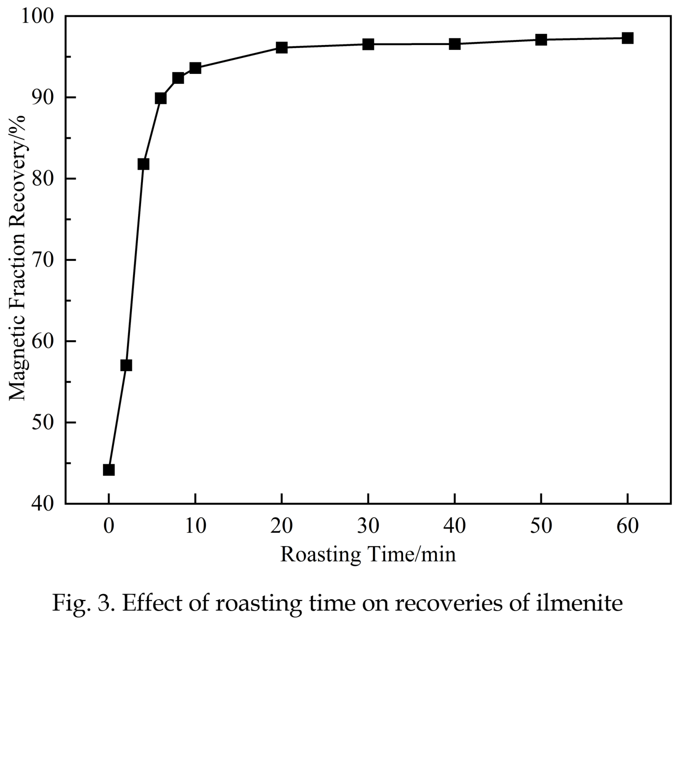

Fig. 3. Effect of roasting time on recoveries of ilmenite

As can be seen from Fig. 3, with the increase in roasting time, the recovery showed a rapid increase and then slowed down until stabilized; when the roasting time was extended from 0 to 20 minutes, the recovery increased from 44.17 to 96.13%; with the further extension of the roasting time, the recovery stabilized at about 96%. Therefore, the roasting time was determined to be 20 minutes.

3.1.3. Effect of cooling method on the results of ilmenite magnetic separation

The cooling method is one of the crucial factors affecting the roasting effect, and the mainstream cooling methods are water quenching, air cooling and furnace cooling. Under the conditions of roasting temperature of 750 °C, roasting time of 20 min, and magnetic field intensity of 238.74 kA/m, the effect of different cooling methods on the behavior of ilmenite oxidation roasting-magnetic separation was investigated, and the results are shown in Fig. 4.

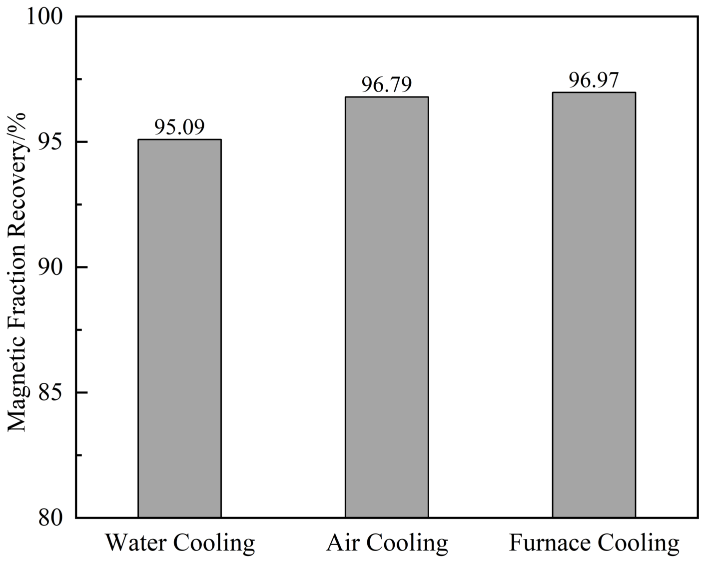

Fig. 4. Effect of cooling method on recoveries of ilmenite

As seen in Fig. 4, the magnetic properties of water-quenched materials are lower than those of air-cooled and furnace-cooled materials. After water quenching, the oxidation reaction stops rapidly due to the absence of oxygen, and the temperature of the material decreases rapidly by heat transfer with water; for air-cooled and furnace-cooled roasted materials, the reaction will be slowly reduced with a slight decrease in the temperature of the roasted material and the oxidation reaction will gradually stop, so that ilmenite is fully transformed into more magnetic minerals and the magnetic properties are increased. This fully indicates that ilmenite's magnetic increase in the atmosphere must have oxygen participation. Rapid cooling (water cooling) may lead to micro-cracks and stress concentrations within the product, damaging its magnetic property and reducing yield. In contrast, slow cooling (air cooling, furnace cooling) helps to maintain structural integrity and magnetic property stability, improving yield. Considering the actual production, determine the cooling method for air cooling, namely, slow cooling.

3.1.4. Effect of magnetic field intensity on the results of ilmenite magnetic separation

Under the conditions of roasting temperature of 750 °C, roasting time of 20 min, and cooling method of air cooling, ilmenite was oxidation roasted. The magnetic separation field conditions were tested before and after ilmenite roasting to investigate the effect of different magnetic field intensities on the magnetic separation behavior, and the test results are shown in Fig. 5.

As can be seen in Fig. 5, the recovery before roasting increases significantly with the increase of magnetic field intensity. The recovery after roasting increased significantly compared to before roasting, and a higher recovery could be achieved at a lower field intensity. With the increase of magnetic field intensity, the recovery of roasted ilmenite firstly increased and then stabilized at about 96%, which further proved that oxidation roasting could enhance the magnetic properties of ilmenite.

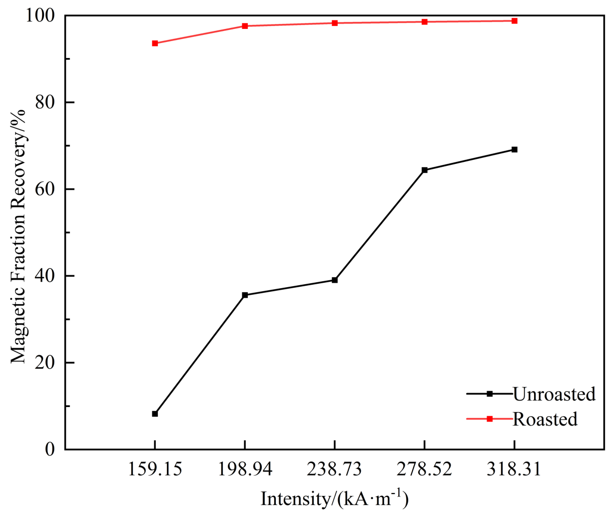

Fig. 5. Effect of magnetic field intensity on recoveries of ilmenite

3.2. Mechanism analysis of phase transformation and magnetic enhancement of ilmenite during oxidation roasting

The previous results of the oxidation roasting-magnetic separation test showed that the magnetic property of ilmenite increased significantly after oxidation roasting due to the phase transformation of ilmenite during the roasting process. Therefore, to find out the mechanism of phase transformation and magnetic properties enhanced, the physical phase composition, magnetic properties and microstructure of the roasted samples were analyzed by XRD, VSM and SEM-EDS under different roasting temperatures, roasting time of 20 min and cooling method of air cooling.

3.2.1. Phase transformation during oxidation roasting

The XRD patterns of the roasted samples at different roasting temperatures are shown in Fig. 6. As the roasting temperature increases, the ilmenite phase gradually transforms into hematite and rutile phases, indicating that ilmenite is oxidized with oxygen in the air to form hematite and rutile (Fig. 6); with the further increase of roast temperature, the pseudobrookite phase gradually increased, indicating that hematite and rutile further reacted to generate pseudobrookite, the magnetic properties of this phase are very weak, which directly led to the rapid decline in the yield of magnetic separation at this time; when the roasting temperature rises to 950 °C, pseudobrookite becomes the most dominant phase.

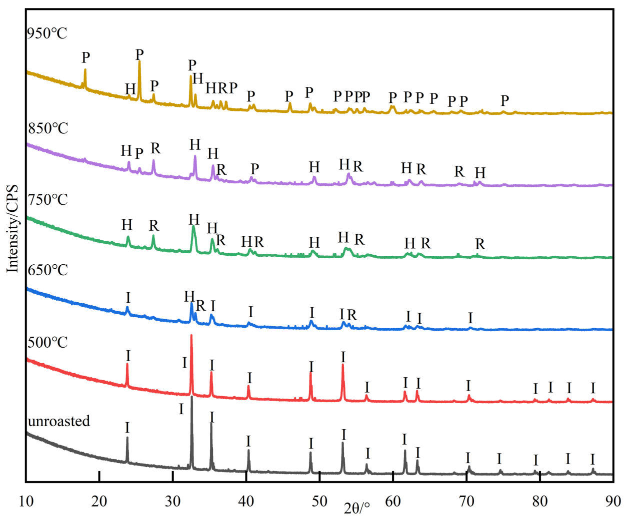

Fig. 6. XRD patterns of roasted ilmenite under different temperatures (I: ilmenite, H: hematite, R: rutile, P: pseudobrookite)

3.2.2. Magnetic properties transformation during oxidation roasting

The results of the magnetic analysis of the roasted samples at different roasting temperatures are shown in Fig. 7. Maximum specific susceptibility of roasted products under different temperatures and corresponding parameters are shown in Table 2.

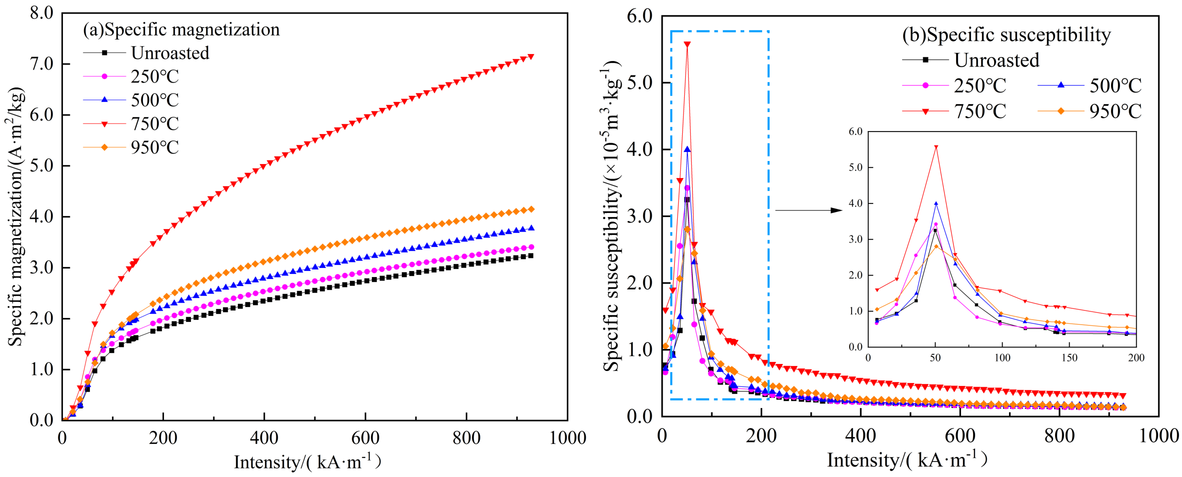

Fig. 7. Magnetic curve of the product under different roasting temperatures. (a) effect on specific magnetization, (b) effect on specific susceptibility

Table 2. Maximum specific susceptibility of roasted products under different temperatures and corresponding parameters

Roasting Temperature /°C	specific susceptibility /($\times 10^{-5} m^3 \cdot kg^{-1}$)	magnetic field intensity /($kA \cdot m^{-1}$)	specific magnetization /($Am^2 \cdot kg^{-1}$)
Unroasted	3.25	49.97	0.61
250	3.42	50.53	0.86
500	3.99	50.61	0.70
750	5.58	50.69	1.32
950	2.81	50.77	0.76

As can be seen in Fig. 7 and Table 2, the magnetic curves of different roasting products have similar characteristics: with the increase of magnetic field intensity, the specific magnetization of the roasting products increases, but the growth rate is gradually slowing down; the specific susceptibility shows a tendency to rise and then decrease and then tends to be stable. However, the specific magnetization and specific susceptibility of different roasting products under the same magnetic field conditions are different. With the increase in roasting temperature, the magnetic properties of roasting products increase first and then decrease. At 750 °C, the main phases are hematite and rutile. Most of the Fe^{2+}in ilmenite is oxidized to Fe^{3+}, forming a ferrimagnet (Deshen and Yixin, 1985). The magnetic property reaches the strongest, and the maximum specific susceptibility of the roasted product increases from 3.25×10^{-5} m^3/kg to 5.58×10^{-5} m^3/kg. Continuing to increase the roasting temperature, hematite and rutile further reacted to form pseudobrookite, the magnetic property weakened, and the maximum specific susceptibility fell back to 2.81×10^{-5} m^3/kg.

3.2.3. Surface microstructural changes during oxidation roasting

The results of SEM-EDS analysis of roasted samples at different roasting temperatures are shown in Fig. 8.

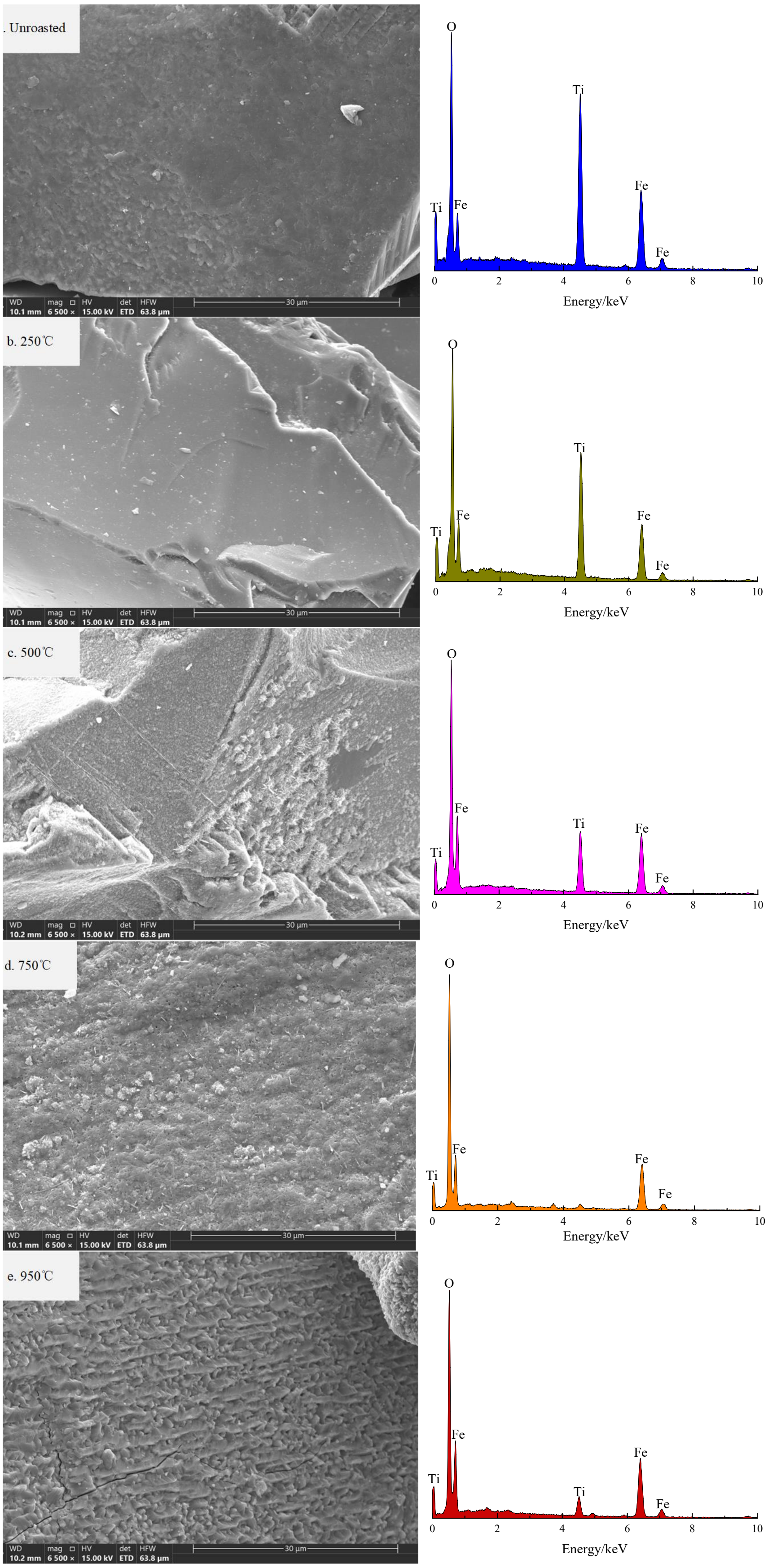

Fig. 8. Scanning electron microscopy images and energy spectra of roasted products under different roasting temperatures

As shown in Fig. 8, ilmenite has a uniform texture, no apparent cracks, and high porosity, and elemental analysis indicates that Fe and Ti exist in the same physical phase of ilmenite at this time. With the increase of roasting temperature, the surface of the particles becomes looser and rougher, the surface structure is damaged, and granular material is gradually formed on the surface of ilmenite because Fe^{2+} on the ilmenite surface is steadily oxidized to Fe^{3+}. At a roasting temperature of 750 °C, the granular material completely covers the surface of the ore. The elemental analysis of EDS spectroscopy found that the Fe element content on the ilmenite surface increased. In contrast, the Ti element decreased, which, combined with the XRD results, indicated that Fe and Ti existed in different phases, suggesting that ilmenite underwent an oxidation reaction and was transformed into hematite and rutile. The surface of the mineral showed fissures at high temperatures, and the surface became rougher, which indicated that ilmenite underwent a violent chemical reaction and physical phase reconstruction at this temperature. EDS spectroscopy analysis was carried out on it, and it was found that Fe and Ti existed in the same physical phase, combined with the XRD physical analysis, the physical phase generated at this time was pseudobrookite, and it was not easy to be captured in the magnetic separation due to its weak magnetic property, and the recovery rate was decreased.

4. Conclusions

This paper studied oxidation roasting-magnetic separation for ilmenite. The effects of different roasting conditions on the oxidation roasting-magnetic separation behavior of ilmenite were analyzed. Suitable roasting conditions were obtained, which resulted in significant enhancement of ilmenite's magnetic properties, and the mechanism of ilmenite's phase transformation and magnetic properties enhancement in the roasting process was analyzed by using XRD, VSM and SEM-EDS, and the main conclusions obtained were as follows:

(1) The mineral magnetic separation test study shows that the magnetic properties of ilmenite roasting products change considerably after oxidation roasting at different conditions. Determine ilmenite suitable oxidation roasting conditions for the roasting temperature of 750 °C, roasting time of 20 min, and cooling method of air cooling; at this time, the yield of 96.79% can be achieved in the magnetic field intensity of 238.74 kA/m.

(2) With the increase of roasting temperature in the oxidation roasting process, ilmenite is transformed into hematite and rutile, enhancing the roasting products' magnetic properties. When the roasting temperature is too high, it will lead to a further reaction between hematite and rutile to generate pseudobrookite, which has a weaker magnetic property and is not beneficial to the recovery of magnetic separation.

Acknowledgments

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