Short Communication

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Change in Microwave-Absorbing Characteristics during the Oxidation Processes of an Ilmenite Concentrate

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Abstract: Microwave-absorbing characteristics of the oxidized ilmenite concentrate were measured by the method of microwave cavity perturbation. The effects of particle size, oxidation temperature and oxidation time on the microwave-absorbing characteristics were investigated. The particle size, oxidation temperature and oxidation time have substantial impact on the microwave-absorbing characteristics of the sample and therefore the microwave heating performance during the oxidation processes of the ilmenite concentrate. Results indicated that at the same oxidation time, the microwave absorbing characteristics decreased as the oxidation temperature increased, and at a constant temperature, the microwave absorbing characteristics of the sample decreased as the oxidation time increased. The microwave absorbing characteristics of the ilmenite concentrate with 80–120 mesh particle size was stronger than that of 200 mesh particle size. The microwave absorbing characteristics of products oxidized at 900 °C for 30 min were slightly weaker than those treated at 800 °C for 30 min and 900 °C for 20 min. The sample becomes less efficient in absorbing microwave energy as the oxidation proceeds. It is therefore recommended strong microwave absorbing materials or conventional heating be applied at the late stage of oxidation to aid microwave heating.

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Introduction

Over the past decade, a number of potential applications of microwave heating in the mineral and materials processing have been investigated, such as microwave assisted sintering, microwave assisted ore grinding, microwave assisted carbothermic reduction of metal oxides, microwave assisted drying and anhydration, microwave assisted mineral leaching, microwave assisted roasting and smelting of sulphide concentrate, microwave assisted pretreatment of refractory gold concentrate, microwave assisted spent carbon regeneration, coke making and activated carbon production, and microwave assisted waste management, solid state synthesis of inorganic materials and preparation of inorganic nanostructures in liquid phase, etc. [1–16]. In 1999, Haque [5] reviewed the application of microwave energy for minerals processing, and pointed out challenges for applications being overcome through a fundamental understanding of microwave interaction with minerals, innovations, R&D investigations and advanced engineering, especially in designing efficient applicator, processes and process control devices. In 2006, Kingman [6] presented a review focusing on the use of microwave heating technologies to improve the efficiency of various mineral processing unit operations (leaching, refractory gold ore treatment, and coal grinding), and opportunities for the future development of microwave technologies for the minerals industry. In 2009, Pickles [10, 11] reviewed the application of microwaves in extractive metallurgy, discussing fundamentals of microwave energy and the utilization of microwave as an energy source in metal extraction, in particular the pyrometallurgical processing of oxide ores. Recently, Peng and Hwang [17] have reviewed the use of microwave energy in extractive metallurgy, with particular emphasis on both fundamentals of microwave heating and recent experimental efforts. Advantages in utilizing microwave technologies for processing minerals and materials include penetrating radiation, controlled electric field distribution, selective and volumetric heating [17]. All these reviews came to the same conclusion that future research should be focused on a combination of reactor design, electromagnetic measurement and simulation, ensuring that the full benefits of microwave heating can be realized. These reviews also emphasize the imperativeness to investigate the microwave absorbing characteristics of materials and minerals. However, there is still little information on the microwave absorbing characteristics of materials and minerals, resulting in difficulties in investigating the interaction mechanism between microwaves and materials, which limits the application of microwave heating in the industry, especially in the chemical and metallurgical industry [17]. Therefore, there is an urgent need to investigate microwave-absorbing characteristics of materials and minerals in detail and collect their dielectric properties, in order to prompt applications of microwave heating in all different kinds of fields.

For the microwave processing of ilmenite, Itoh et al. studied the microwave oxidation of an ilmenite ore to produce rutile [18]. Kelly and Rowson investigated the microwave reduction of the oxidized ilimenite concentrate [19]. Tong et al. evaluated the economic advantages of industrial applications of carbothermic reduction of metal oxides by microwave heating, showing that application of microwave heating reduced operating costs by 15–50 % compared to that of conventional method [20]. Cutmore et al. investigated dielectric properties of some minerals [21].

The authors' group has recently investigated the carbothermic reduction kinetics of ilmenite concentrate using sodium silicate, sodium chloride, and sodium borate as catalysts, respectively, and microwave-absorbing characteristics of ilmenite concentrate containing different proportions of carbonaceous reducing agents and reduced products [22–26]. This has allowed us to optimize the ilmenite concentrate processing and microwave cavity design.

The objective of the present study was to further investigate the effect of the particle size, oxidation temperature, and the oxidation time on the microwave-absorbing characteristics of the oxidized ilmenite concentrate in order to fully utilize microwave technology in the ilmenite concentrate processing.

Experimental

Materials

The ilmenite concentrate was obtained from Panzhihua (Sichuan province, PR China). The main chemical composition of the ilmenite concentrate was listed in Table 1.

Table 1: Chemical composition of the ilmenite concentrate (%).

Chemical compositions	TFe	TiO_2	CaO	MgO	SiO_2	Al_2O_3	S
Content (wt%)	32.18	47.85	1.56	6.56	5.6	3.16	≤ 0.1

Measuring principles of microwave absorbing characteristics

The principle and equipment for measuring the microwave absorbing characteristics of resulting products using the microwave cavity perturbation method can be found in our previously published papers and patent [22, 27, 28].

The equipment in the present study consists of a resonant sensor [28], sweeping signal, detector and DSP (Digital Signal Processors), interface circuit and computer. It was controlled by a software programmed in Visual Basic 6.0.

Cavity perturbation technique was widely adopted for microwave dielectric properties measurements, which is based on the fact that cavities are high-quality resonance structures [29]. Coupling between microwaves and materials is determined by electrical permittivity and magnetic permeability. The presence of a small piece of dielectric sample in the resonant cavity will cause a shift of resonant frequency and a decrease of the quality factor of the cavity. The decrease in the quality factor of the cavity is because of the presence of sample's dielectric loss. The dielectric constant and loss tangent of the specimen can then be calculated from the changes of resonance frequency and quality factor. In a word, the success of cavity perturbation method to calculate the microwave dielectric properties relies on measuring the values of resonant frequency and quality factor accurately, before and after the insertion of the sample into the cavity. The cavity is calibrated with dimensionally identical sample of known permittivity.

Each material has intrinsic properties relative to the absorption of microwave energy. The most important property is the dielectric constant, or permittivity, ε. The permittivity is a measure of a material's ability to absorb and to store electrical potential energy. The measurement principle is that microwave is coupled with the microwave cavity resonator, that is to say, is the measurement of resonant frequency and the output voltage of the resonant sensor unloaded and loaded with samples. In this cavity, microwave interacts with the sample measured. When the sample is very small (the volume of the sample is much smaller than the volume of the cavity), the technique of perturbation can be applied [22, 27, 29].

$$\frac{\Delta\omega}{\omega} = -\omega_0(\varepsilon_r' - 1)\int_{Ve} E_0^{\star} \cdot EdV/4W \quad (1)$$

$$\frac{1}{Q} - \frac{1}{Q_0} = 2\varepsilon_0\varepsilon_r''\int_{Ve} E_0^{\star} \cdot EdV/4W \quad (2)$$

$$W = \int_V [(E_0^{\star} \cdot D_0 + H_0^{\star} \cdot B_0) + (E_o^{\star} \cdot D_1 + H_0^{\star} \cdot B_1)]dV \quad (3)$$

where ω is the angular frequency, ω_0 is the angular frequency without sample in resonant cavity, $\Delta\omega = \omega - \omega_0$, is the shift of angular frequency. E is the electric field intensity of the sample in the resonant sensor, $E_0^{\star}$,$H_0^{\star}$ are the hetero conjugations of electric field intensity and electromagnetic field intensity in the resonant sensor before perturbation, respectively. D_0, and B_0 are the hetero conjugations of electric displacement and magnetic induction before perturbation, respectively, while D_1 and B_1 are the increments of electric displacement and magnetic induction in samples after perturbation. V is the volume of the resonant sensor, V_e is the volume of the sample in the resonant sensor, d_v is the volume of the element. Q_0 and Q are the quality factors (Q values) of the cavity unloaded (unperturbed condition) and loaded with the samples, respectively. ε_o is the absolute permittivity of a vacuum (free space), relative permittivity ε_r'' and relative loss factor ε_r are the real and the imaginary part of the complex permittivity or dielectric loss of the sample, respectively. ε_r'' is a measure of the penetration of microwaves into the material, in most cases, it is not a constant but it is a strong function of both the temperature and microwave frequency (for vacuum $\varepsilon_r' = 1$). ε_r is a measure of the material's ability to store the energy and will also be dependent on the temperature and microwave frequency. That is to say, both the relative permittivity ε_r' and the relative loss factorε_r are functions of frequency, temperature, and material properties. The frequency dependence of ε_r and ε_r' and their magnitudes control the extent to which a substance is able to couple with microwave radiation. The ratio of ε_r'' to ε_r' is called the (dielectric) loss tangent (tan δ). The substance is lossless if $\varepsilon_r'' = 0$. W is the storage energy of resonant sensor. The resonant cavity mode is TM_{010} (80 mm inner diameter, 12 mm height), the inner diameter of the sample chamber of the cavity is 5 mm, and the sample was distributed uniformly in the resonant cavity. A vector network analyzer (HP9000/300) is used to detect the frequency shift, and resultant quality factor. The whole measurement and network analyzer analysis were all controlled by a computer. In order to further minimize the error, the cavity was first calibrated with alumina and silica with their size and shape similar to the sample.

By using eqs (1)–(3), microwave absorbing characteristics of materials can be obtained through inversion by measuring variations of microwave output and resonant frequency of the resonant sensor unloaded and loaded with samples.

Experimental setup and conditions

Microwave heating setup (3 kW, frequency 2.45 GHz) was illustrated in Figure 1 which was researched and developed by the authors' group. A microwave system typically consists of a generator (magnetron) to produce the microwaves, a waveguide to transport the microwaves, and applicator (usually a cavity) to manipulate microwaves for a specific purpose, and a control system (tuning, temperature, power, etc).

Oxidation of ilmenite concentrate was performed in the reaction cavity with an air flow continuously blown into the microwave heating setup. The oxidation process of the ilmenite concentrate was carried out at the temperature of 700–900 °C for the oxidation time of 10–30 min with the particle size range of 80–200 mesh.

XRD characterization

XRD data were obtained using X-ray powder diffractometer (D/max-2000; Rigaku, Tokyo, Japan) in the diffraction angle range 2θ = 20–60°, using Cu $K_\alpha$radiation.

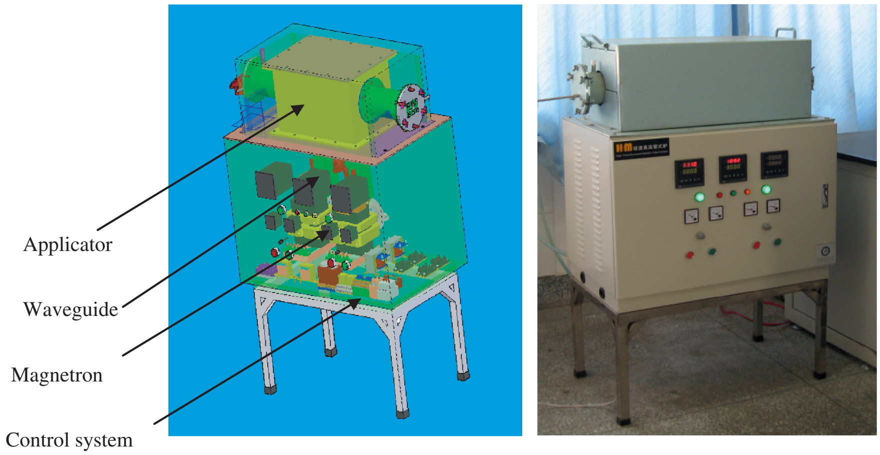

Figure 1: Schematic diagram (left) and photo (right) of the microwave heating apparatus.

Results and discussion

Microwave absorbing characteristics of the oxidation products of the ilmenite concentrate.

Although the oxidation process of the ilmenite concentrates by conventional heating has been extensively investigated up to now, there is still little research regarding the oxidation process of the ilmenite concentrate by microwave heating. The study of the microwave absorbing characteristics of the oxidation products is favorable to the utilization of microwave technology for the oxidation process of the ilmenite concentrate. If the oxidation products of the ilmenite concentrate were microwave absorbing materials, the absorbing efficiency of microwave energy during oxidation would be enhanced, otherwise, the oxidation process should be assisted by adding the strong microwave absorbing material, such as carbon, to increase the absorbing efficiency of microwave energy. Therefore, the microwave absorbing characteristics of the oxidation products of the ilmenite concentrate oxidized under different conditions were measured by the method of microwave cavity perturbation [22, 27, 29]. The mechanism for this technique is the measurement of resonant frequency and the output voltage of the resonant sensor unloaded and loaded with samples. By using eqs (1)–(3), the microwave absorbing characteristics of the samples can be obtained by measuring variations of microwave voltage output and resonant frequency of the resonant sensor unloaded and loaded with samples.

Figure 2 shows the microwave spectra of the oxidation products oxidized from the ilmenite concentrate of varying sizes at different temperatures for increasing time (The lines not labeled with time are the spectra for raw samples). It can be seen that the microwave absorbing characteristics of the oxidation products of the ilmenite concentrate are different, demonstrating that the particle size, oxidation temperature and oxidation time have substantial impact on the microwave-absorbing characteristics of oxidation products during the oxidation processes of the ilmenite concentrate. From these microwave spectra, the parameters of attenuation (microwave voltage output) and microwave frequency shift (compared to the fixed frequency, 2.45 GHz) can be obtained, and used to evaluate the effects of the particle size, temperature and time on the microwave-absorbing characteristics of oxidation products during the oxidation processes of the ilmenite concentrate.

The effect of the concentrate size on the microwave absorbing characteristics of oxidation products of ilmenite concentrate

Figure 3 shows the relationship between the attenuation/microwave frequency and the concentrate size during the oxidation process (oxidation temperature 700 °C, oxidation time 30 min) of ilmenite concentrate with different particle size. A weak microwave absorbing characteristics material, TiO_2, was included for comparison. It can be found that the voltage attenuation in the microwave resonator is the smallest for particle size of −80 ~ +120 mesh, while the microwave frequency shift is the largest,

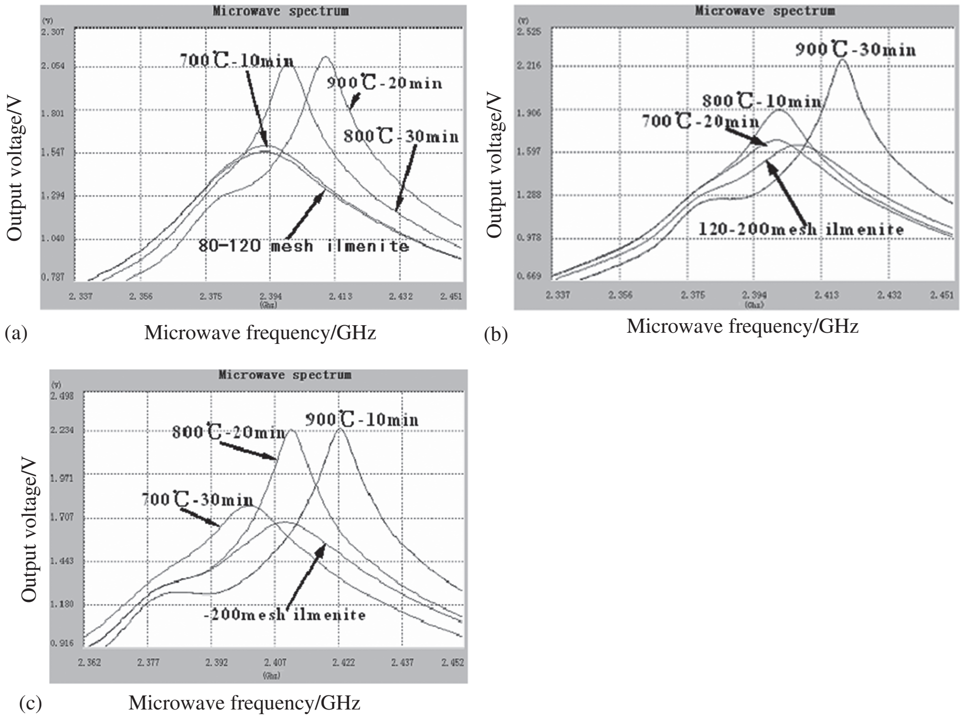

Figure 2: The microwave spectra of oxidation products oxidised at different temperatures for varying times from the ilmenite concentrates of different sizes: (a) particle size: −80 +120 mesh; (b) particle size: −120 +200 mesh; (c) particle size: −200 mesh. The lines not labeled with time are the spectra for raw samples.

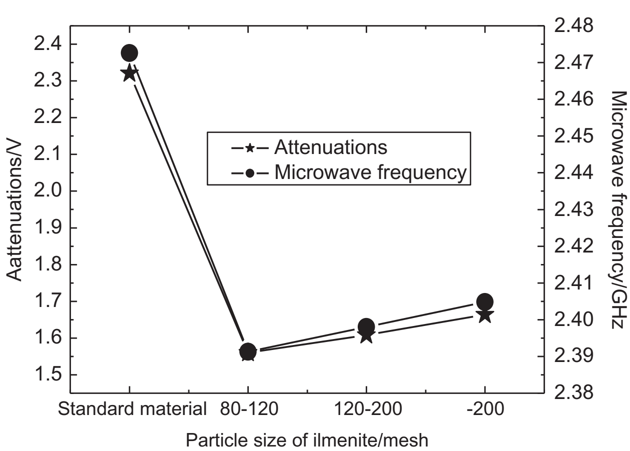

Figure 3: Effect of ilmenite concentrate size on the attenuation and microwave frequency of oxidation products (oxidation temperature is 700 °C and oxidation time is 30 min) (Standard material: TiO_2 with its particle size of −40 + 80 mesh).

(ε_r is a measure of the material's ability to store the energy, which is in inverse proportion to the voltage attenuation. $\varepsilon' - 1$ is in proportion to the microwave frequency shift, ε_r' is a measure of the penetration of microwaves into the material, the ratio of ε_r'' to ε_r' is called the (dielectric) loss tangent (tan δ), which is the indicative of the ability of microwave heating for a specific material at a given frequency and temperature). Therefore, the particle size range of −80 ~ +120 mesh showing the highest microwave absorbing characteristics was selected.

It is well documented that the depth of penetration of the microwave radiation (being defined as the depth at which the microwave power has fallen to 1/e of its incident value) is also an important factor in dielectric heating, because an even distribution of microwave in a material is essential for the uniform heating [1, 6, 14, 17]. The penetration depth (D_P) can be calculated by using the following equation.

$$D_p = \frac{\lambda_0}{2\pi(2\varepsilon_r)^{1/2}} \left\{ \left[1 + \left(\frac{\varepsilon_r''}{\varepsilon_r'} \right)^2 \right]^{1/2} - 1 \right\}^{-1/2} \quad (4)$$

where D_P is the penetration depth; λ_O is the free space wavelength of incident microwave.

The properties that most strongly influence the penetration depth are the dielectric properties of the material and the wavelength of microwave. If a material has a small penetration depth, then all of the power will be lost near the surface of the material. High frequencies and large values of the dielectric properties will result in surface heating, while low frequencies and small values of dielectric properties will result in more volumetric heating. The penetration depth (D_P) shows the degree of interaction between microwaves and materials. Therefore it is a very important factor in the design and scale up of a microwave heating system.

Roy et al. [2] carried out the full sintering of metal bodies in a microwave field, concluding that the shape and size of metals play an important role in the sintering process by microwaves. Lekse et al. [3] investigated the synthesis of intermetallic compounds via microwave irradiation, indicating that the particle size of materials can change materials' microwave absorbing behavior, even from absorbing to reflecting. For most materials at the range of microwave frequencies, the penetration depth is of the same order of magnitude as the dimensions of the sample, a fact that has important implications for uniform heating. If the volume of the sample is too large, then insufficient microwave energy reached the center of the sample, resulting in inhomogeneous products due to non-uniform heating [1, 6, 15, 17].

The present results also demonstrated that the particle size of materials had great impact on the microwave absorbing characteristics of the materials, even if the materials were microwave-absorbing materials.

The effect of the oxidation temperature on the microwave absorbing characteristics of oxidation products of ilmenite concentrate

It is well known that some materials are weak microwave-absorbing at a lower temperature, and become strong microwave-absorbing when being heated to a certain higher temperature [1, 2, 17]. Figure 4 shows the attenuation/microwave frequency of oxidation products oxidized at 700, 800 and 900 °C for 10, 20 and 30 min, respectively, from 80 to 120 mesh ilmenite concentrate. It can be found that as the oxidation temperature increases, the voltage attenuation increases while the microwave frequency shift decreases although the microwave frequency shift of the oxidation product treated at 700 °C for 20 min is similar to that for the oxidation product treated at 800 °C for 20 min. Based on the impact of the attenuation/microwave frequency shift on the microwave absorbing characteristics, the oxidation product treated at 700 °C for 10 min should have the strongest microwave absorbing characteristics. Results also suggest that at the same oxidation time, the microwave absorbing characteristics generally decreases as the oxidation temperature increases.

In a word, the dielectrics properties of a specific material will vary with temperature. So, it is vital to investigate the variation in dielectrics properties across a broad range of conditions to fully understand the behavior of the material in the microwave field.

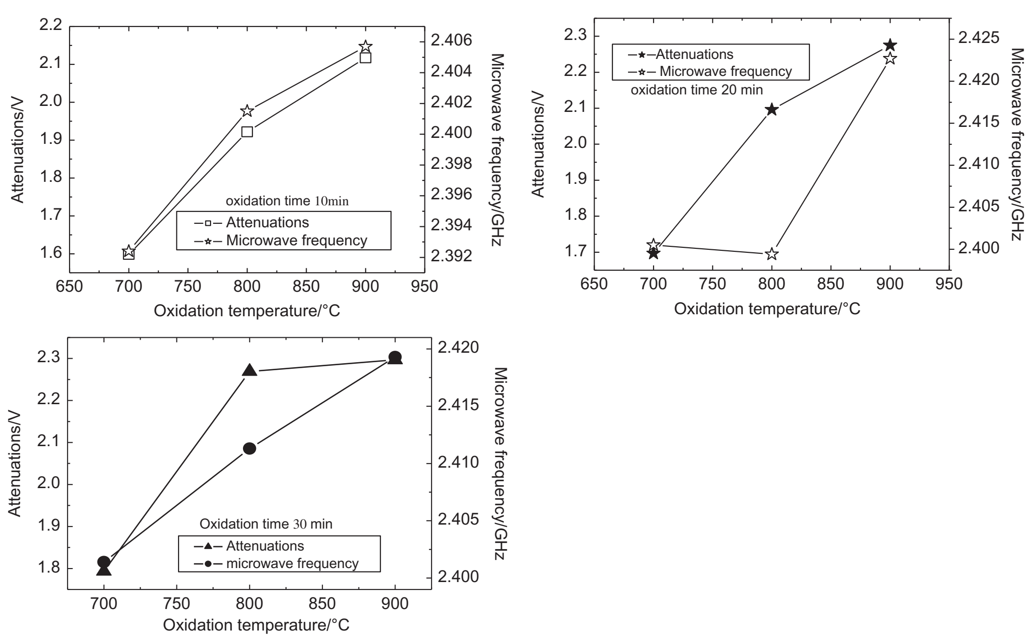

Figure 4: Effect of oxidation temperature on the attenuation and microwave frequency of oxidation products treated for 10 min (top), 20 min (middle) and 30 min (bottom) from the −80 + 120 mesh ilmenite concentrate.

The present results further illustrated that the microwave absorbing characteristics of the oxidized ilmenite concentrate exhibits a strong temperature dependence.

The effect of the oxidation time on the microwave absorbing characteristics of oxidation products of ilmenite concentrate

Figure 5 shows the effect of oxidation time on the attenuation/microwave frequency shift of oxidation products treated at 700, 800 and 900 °C, respectively, from the 80–120 mesh ilmenite concentrate. It can be found that as oxidation time increases, the voltage attenuation increases while the microwave frequency shift generally decreases except for the oxidation products treated at 800 and 900 °C for 20 min. Based on the impact of the attenuation/microwave frequency shift on the microwave absorbing characteristics, it is found that at a constant temperature, the microwave absorbing characteristics of the sample decreases as the oxidation time increases.

It can be found that the attenuation voltage at the processing conditions of 900 °C-30 min, 800 °C-30 min and 900 °C-20 min are 2.2968 V, 2.2749 V and 2.2693 V, respectively, showing that the microwave absorbing characteristics of oxidation products treated at 900 °C for 30 min is slightly weaker than those treated at 800 °C for 30 min and 900 °C for 20 min. So, the product obtained at 900 °C-30 min was characterized by XRD (Figure 6). It is shown that most of the Fe^{2+} in the ilmenite concentrate has been oxidized to Fe^{3+}, forming Fe_2O_3, indicating that the ilmenite concentrate has been oxidized completely with an oxidation condition of 900 °C for 30 min. In addition, rutile and anatase TiO_2 are also observed but no $FeTiO_3$ is present.

Based on the above analysis of microwave absorbing characteristics of products with different oxidation conditions, it is indicated that the sample becomes less efficient in absorbing microwave energy as the oxidation proceeds (the correspondingε' was small), resulting in the increasing energy consumption of microwave heating. It is therefore recommended at the late stage of oxidation to add strong microwave absorbing materials to aid microwave heating or to supplement microwave heating with conventional heating.

In summary, the particle size, oxidation temperature and oxidation time have substantial impact on the microwave-absorbing characteristics of the sample and therefore the microwave heating performance during the oxidation processes of the ilmenite concentrate. The particle size of −80 + 120 mesh of ilmenite concentrate has the best microwave absorbing characteristics, and the microwave absorbing characteristics of the oxidation

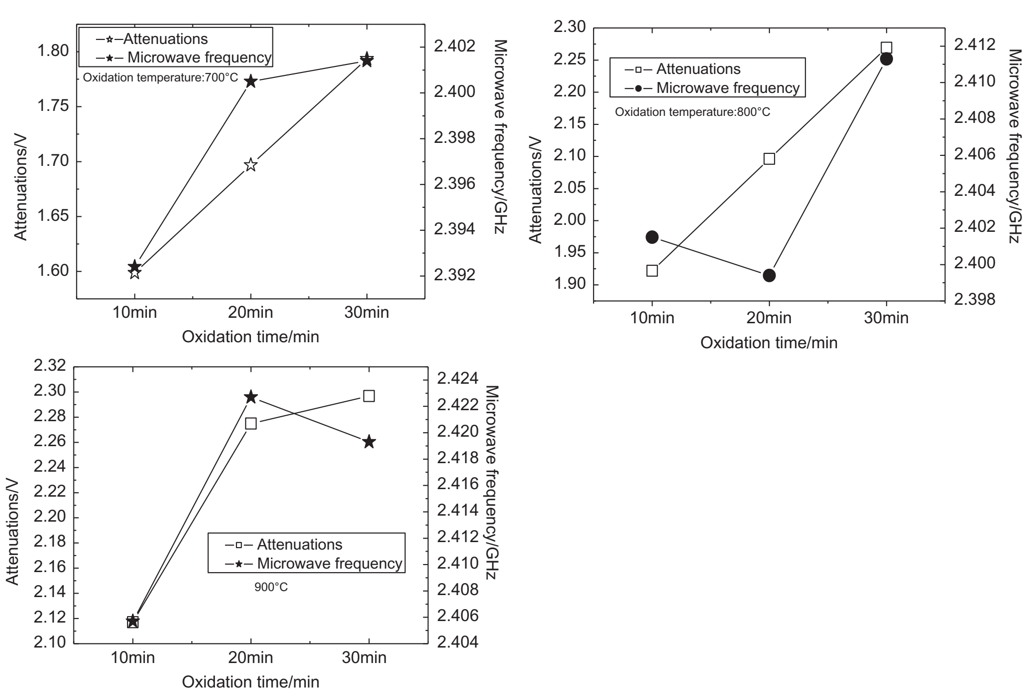

Figure 5: Effect of oxidation time on the attenuation and microwave frequency of oxidation products treated at 700 (top), 800 (middle) and 900 °C (bottom) from the −80 + 120 mesh ilmenite concentrate.

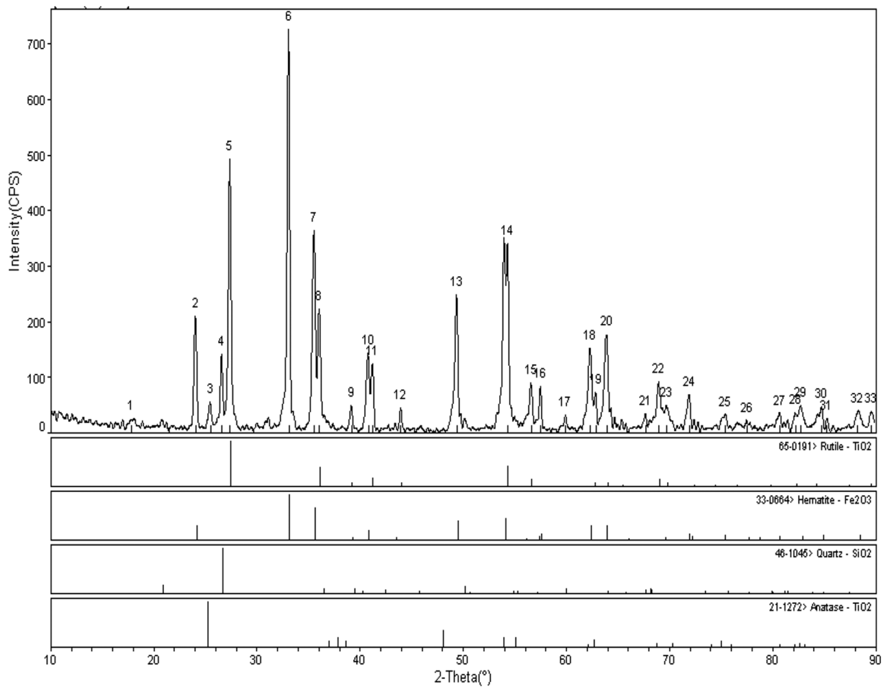

Figure 6: The XRD pattern of the oxidation product treated at 900 °C for 30 min from −80 + 120 mesh ilmenite.

products treated at 900 °C for 30 min is slightly weaker than those treated at 800 °C for 30 min and 900 °C for 20 min, and the microwave absorbing characteristics of the oxidation products of the ilmenite concentrate exhibits a strong temperature dependence.

Conclusions

The present results demonstrated that the particle size, oxidation temperature and oxidation time had substantial impact on the microwave-absorbing characteristics of the sample and therefore the microwave heating performance during the oxidation processes of the ilmenite concentrate. The microwave absorbing characteristics of the ilmenite concentrate with 80–120 mesh particle size was stronger than that of 200 mesh particle size. The microwave absorbing characteristics of the oxidized products of the ilmenite concentrate exhibits a strong temperature and time dependence. Results indicated that at the same oxidation time, the microwave absorbing characteristics decreases as the oxidation temperature increases, and at a constant temperature, the microwave absorbing characteristics of the sample decreases as the oxidation time increases. The sample becomes less efficient in absorbing microwave energy as the oxidation proceeds (the corresponding ε' was small), resulting in the increasing energy consumption of microwave heating. It is therefore recommended at the late stage of oxidation to add strong microwave absorbing materials to aid microwave heating or to supplement microwave heating with conventional heating.

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