

Thermally Assisted Grinding of Cassiterite Associated with Polymetallic Ore: A Comparison between Microwave and Conventional Furnaces

Chunlin He ^{1,2,*}, Jian Zhao ^{1,2}, Xiujuan Su ^{1,*}, Shaojian Ma ^{1,2}, Toyohisa Fujita ^{1,2}, Yuezhou Wei ^{1,2,3}, Jinlin Yang ^{1,2} and Zongwu Wei ^{1,2}

¹ School of Resources, Environment and Materials, Guangxi University, Nanning 530004, China; ZJ19780823@163.com (J.Z.); msj@gxu.edu.cn (S.M.); fujitatoyohisa@gxu.edu.cn (T.F.); yzwei@gxu.edu.cn (Y.W.); 1615391004@st.gxu.edu.cn (J.Y.); fangshen@gxu.edu.cn (Z.W.)

² Guangxi Key Laboratory of Processing for Non-Ferrous Metal and Featured Materials, Nanning 530004, China

³ School of Nuclear Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, China

* Correspondence: helink@gxu.edu.cn (C.H.); zhsxty@gxu.edu.cn (X.S.)

2. Materials, Equipment and Methods

2.2. Equipment and Methods

(1) Multiple Materials Discretely Coexisted

Various materials were evenly placed around the rotary table, as Figure S1 shows. After microwave heating for a certain time, the surface temperature of all materials was tested by infrared imager (Wuhan Guide infrared TP80s). The infrared imager can easily and quickly compare the heating effect of different materials after microwave heating. Considering that the electromagnetic parameters of some substances may change under high temperature, the temperature was controlled at a lower temperature. Microwave heating was conducted in a 2450 MHz microwave oven (volume 80 L), and the power of microwave was set at 1 kW for 60 s.

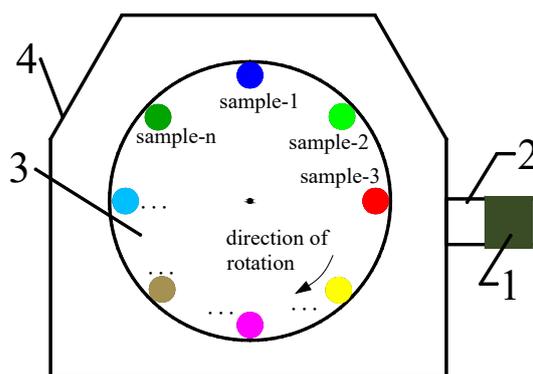


Figure S1. Schematic illustration of multiple materials discretely coexisted. 1. Magnetron, 2. Waveguide, 3. Turntable, 4. Microwave cavity.

(2) Heating Rates

Microwave heating was conducted in a 2450 MHz microwave oven (volume 80 L), as shown in Figure S2. Each sample (100 g) was placed in a quartz crucible (100 mL). The microwave output power was set at 1 kW and a temperature measurement was conducted using a metal sheathed thermocouple, which was in constant contact with the sample, so the temperature could be recorded continually. The temperature was measured in the center of the sample and the sample was placed in the sample place for each experiment.

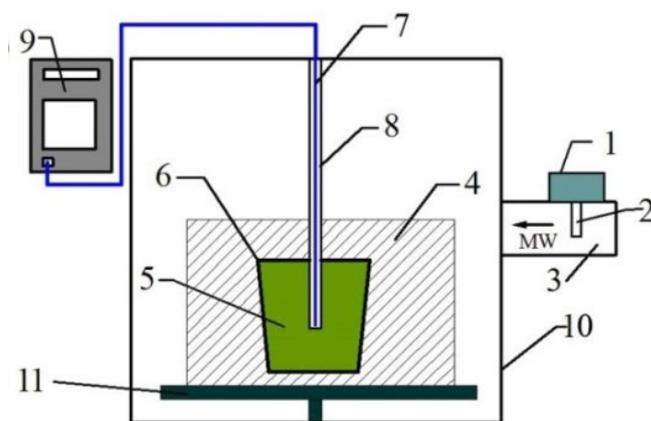


Figure S2. Microwave apparatus for heating curves. 1. Magnetron, 2. Antenna cap, 3. Waveguide, 4. Thermal insulation, 5. Sample, 6. Quartz crucible, 7. Thermocouple, 8. Metal shielding tube, 9. Digital thermometer, 10. Microwave cavity, 11. Turntable.

(3) Calorimetry Measurement

The ability of the different materials to absorb microwaves was measured using a household microwave oven (EV025LC7-NR) with microwave output power level of 100–1000 W to measure the quantitative microwave energy absorption of different materials as shown in Figure S3. The ability of the microwave absorption characteristics was compared by the relative energy (RE). Water was used as the medium material for our experiments. Water is sensitive to microwaves and is mixed easily so that its inner temperature remains constant for convenient temperature measurements. Water and the tested materials were placed in the same cavity to absorb microwaves. If water does not undergo a phase change, the variation of its inner temperature should reflect its microwave absorption. An electric thermocouple probe was inserted into the water medium only after the oven power had been turned off and the water medium was shaken lightly to even out the inner temperature. Each test was repeated three times, and the data presented are averages of these tests. The operating conditions were constant for all experiments to enable a comparison of the microwave energy absorption between different materials. Temperature measurements were taken mainly for the water medium. The relevant calculation details are as follow:

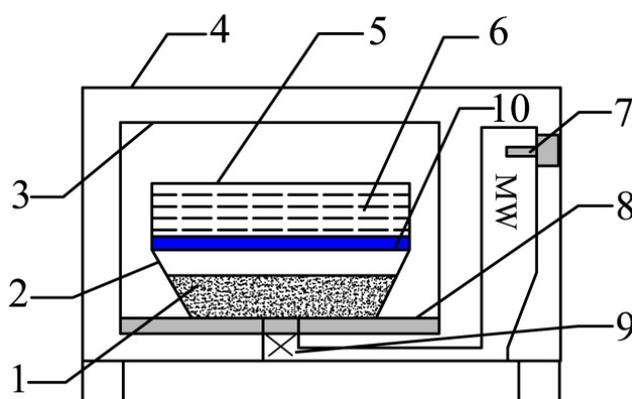


Figure S3. Diagram of measuring apparatus. 1. Test materials, 2. Quartz container, 3. Metal cabinet, 4. Microwave oven, 5. Plastic box, 6. Water medium, 7. Operation panel, 8. Ceramic plate, 9. Microwave outlet of waveguide, 10. Thermal insulation material (quartz fiber).

W_{OM} is the total output microwave energy; Q_{TM} and Q_w are the available microwave energy in the cavity for heating the test material and heating the medium, respectively,

and Q_L is the ineffective loss of microwave energy, which can be treated as a constant. The following equation can be derived:

$$W_{OM} = Q_w + Q_{TM} + Q_L \quad (1)$$

When there is no test material except for the water, then the energy absorbed by the water can be expressed as:

$$q_w = mc(t_2 - t_1) + L(t) \quad (2)$$

where m is the water mass, c is the specific heat capacity of water (4.18 kJ/kg°C), t_1 is the initial water temperature before heating and t_2 is final temperature after heating. When a test material was added, the energy absorbed by the water can be written as:

$$Q_w = mc(T_2 - T_1) + L(T) \quad (3)$$

where T_2 and T_1 are the water temperature after and before heating, respectively. Therefore, the microwave energy absorbed by the test material (Q_{TM}) can be derived as follows:

$$Q_{TM} = q_w - Q_w \quad (4)$$

For the sake of convenient discussion and comparison of the microwave energy absorption of different materials, the RE was used as an assessing index, and can be defined as follows:

$$RE = Q_{TM} / q_w \quad (5)$$

Thus, the microwave energy absorbed by a material can be obtained from Equation (4). Furthermore, the microwave absorption of different materials can be compared using their RE values from Equation (5).

(4) Dielectric Properties

The microwave system for measuring the complex permittivity is shown in Figure S4. The reflection (S_{11} and S_{22}) and transmission (S_{21} and S_{12}) of the electrical signal, termed the scattering parameter, were measured by the vector-network analyzer (Agilent N5244A PNA-X) using a coaxial transmission-reflection method, which has a broadband frequency range. The complex permittivity ($\epsilon = \epsilon' - i\epsilon''$) was determined from the scattering parameters using the Nicholson-Ross-Weir models.

For dielectric property measurements, each test sample was ground to -0.075 mm, and then mixed homogenously with paraffin wax at a mass ratio of 7:3. The wax was used to fix the sample power so it can be loaded into the tested artifacts. The real permittivities of paraffin wax were approximately 2.1 and 1, respectively, and their imaginary permittivities and permeabilities were approximately 0. The mixture samples were pressed into a toroidal-shaped sample of 7.00 mm outer diameter and 3.04 mm inner diameter with a 2.0 mm thickness. In this study, the permittivities were measured from 2 to 18 GHz. The following refers to dielectric property and was the results of mixture samples with 70% target sample fraction.

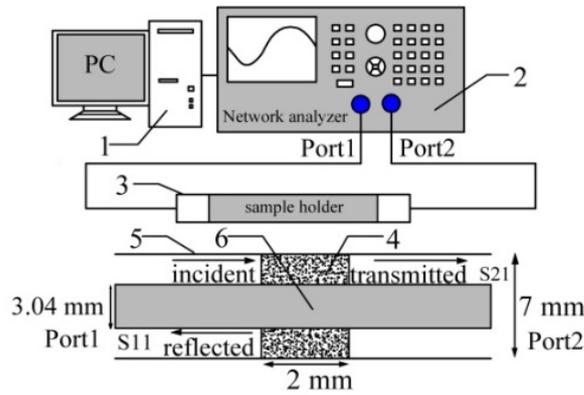


Figure S4. The scheme of magnetic and dielectric properties measurement. 1. Computer, 2. Network analyzer, 3. Coaxial transmission line, 4. Sample, 5. Outer conductor, 6. Inner conductor

3. Ability of Minerals in Absorbing Microwaves

The real (ϵ') and imaginary (ϵ'') permittivities of sample from 2 to 18 GHz are shown in Figure S4. The real and imaginary permittivities exhibit a similar fluctuating behavior with microwave frequency. As can be seen from Figure S4(b), the imaginary permittivities (ϵ'') of pyrite is the highest among the samples, and the ϵ'' of sphalerite exhibits low dielectric constants.

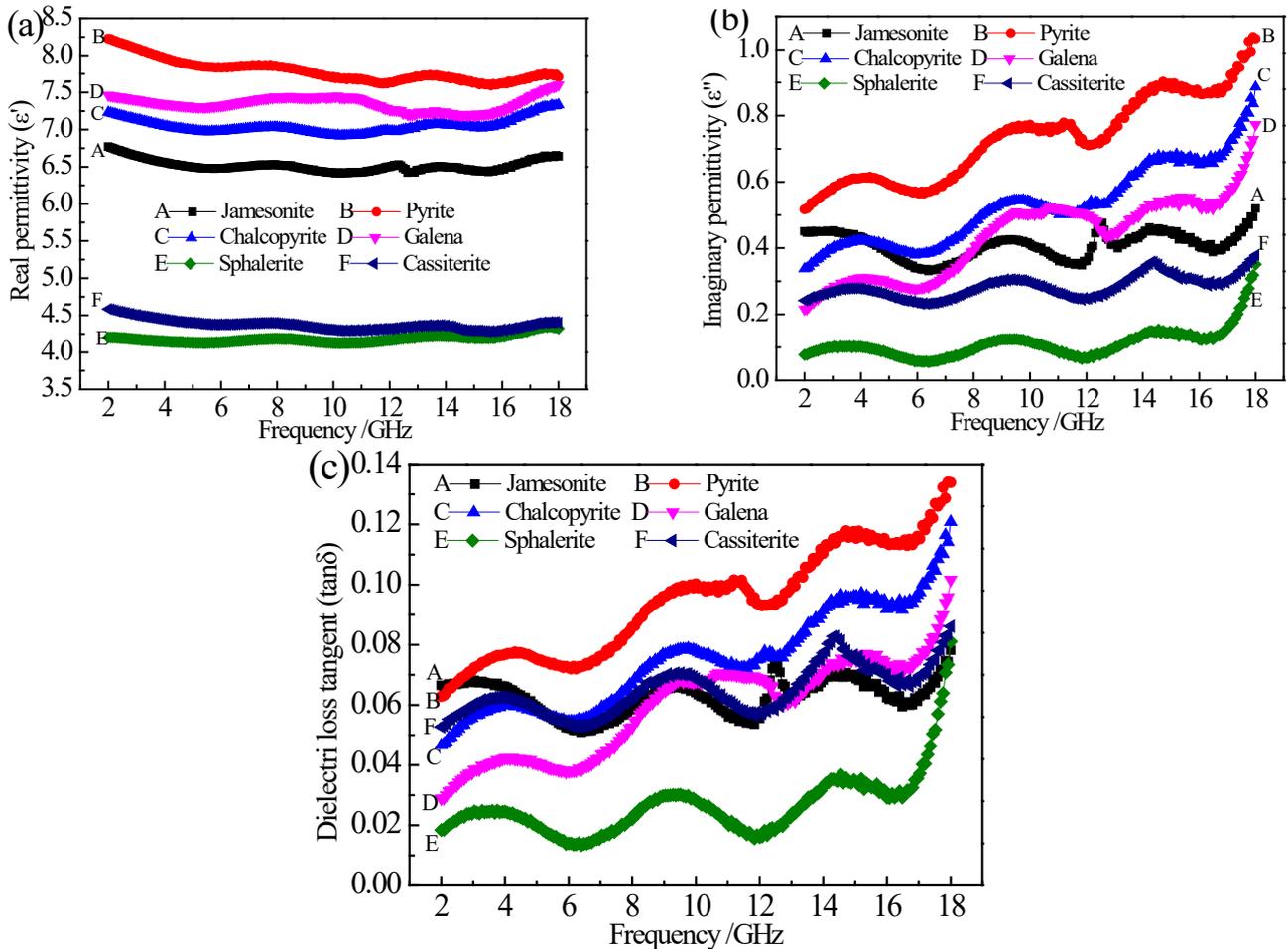


Figure S5. Dielectric properties of the sulphide ore and SnO₂: (a) ϵ' , (b) ϵ'' , (c) $\tan\delta$.

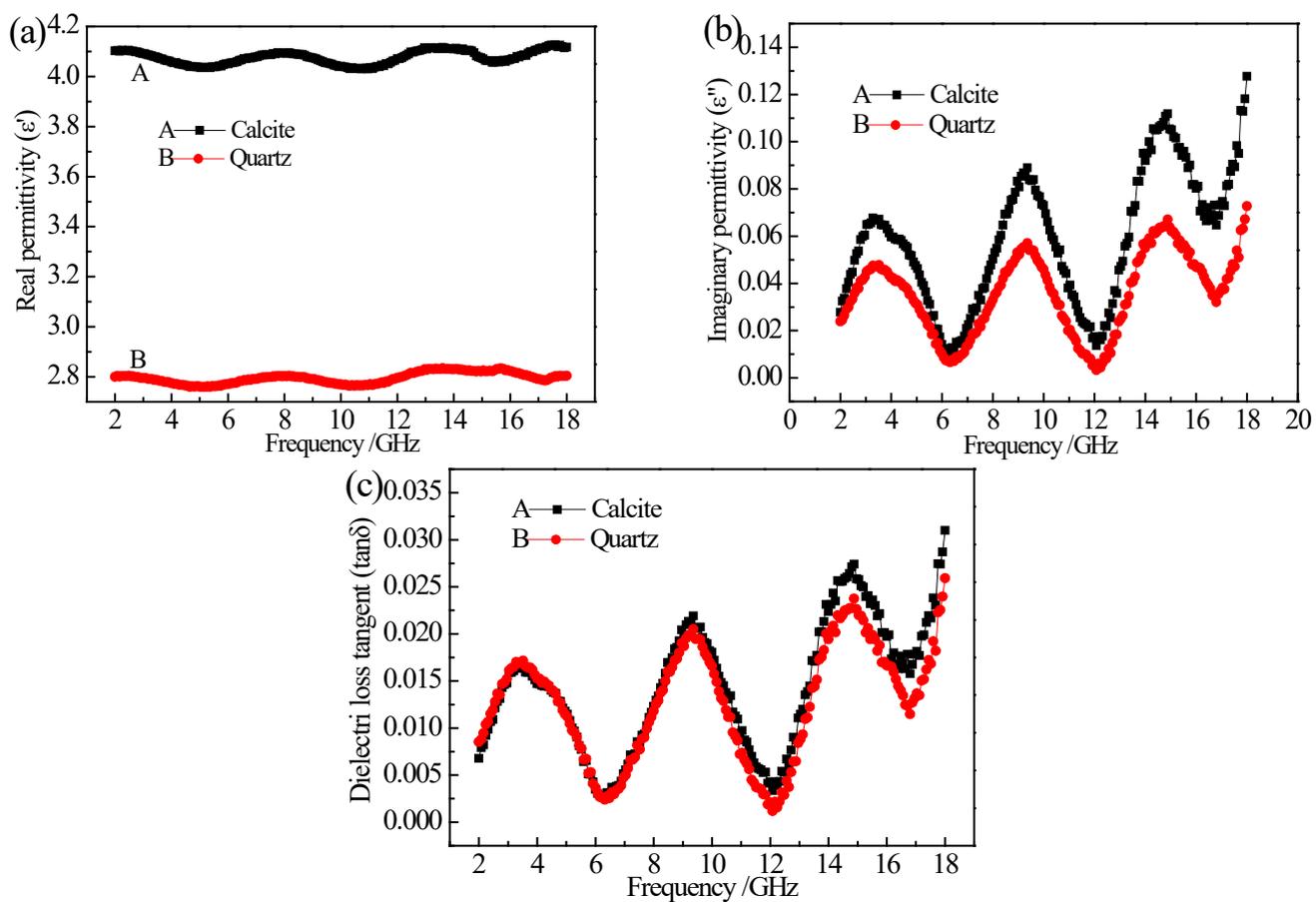


Figure S6. Dielectric properties of calcite and quartz (a) ϵ' , (b) ϵ'' , (c) $\tan\delta$.

4. Grinding test

(2) The effect of microwave heating time on temperature of sample

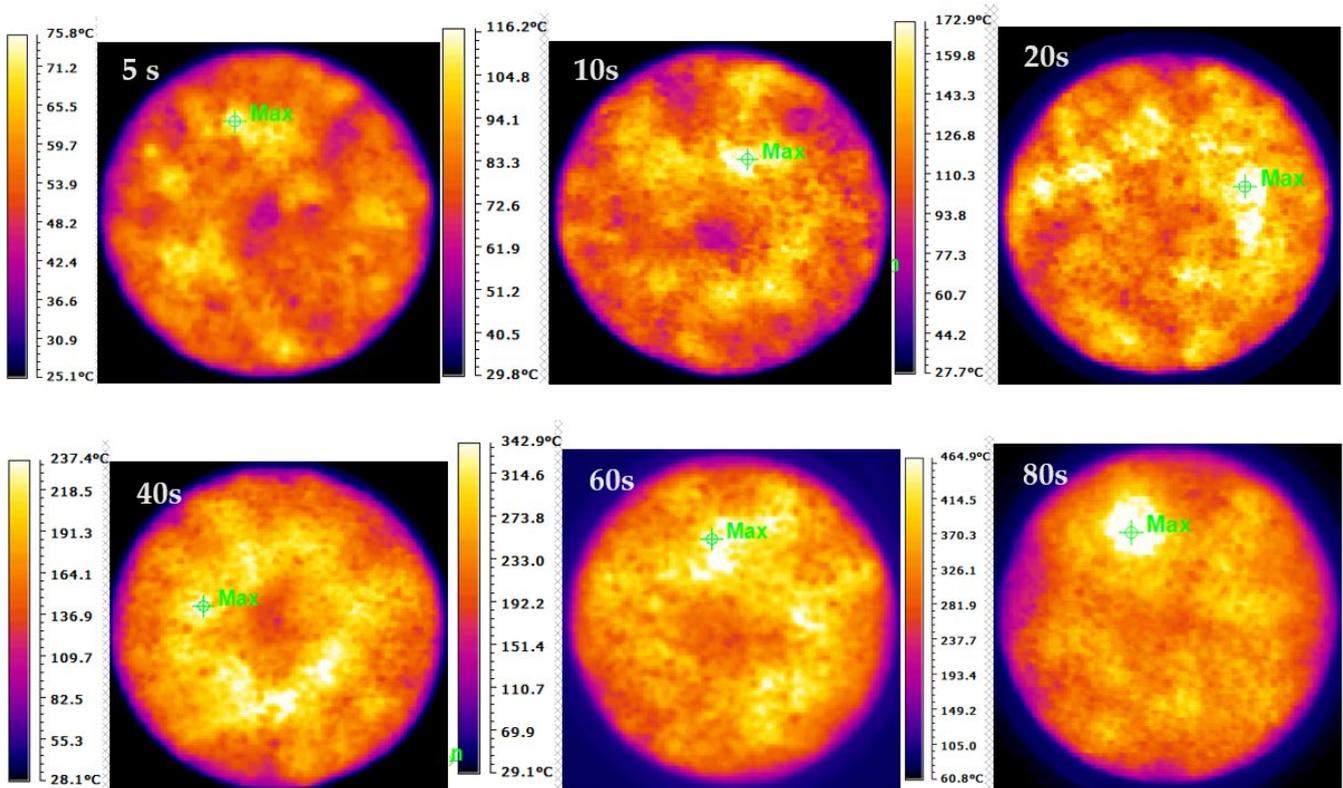


Figure S7. Effect of microwave heating time on distribution of surface temperature of test sample.

Table S1. Effect of microwave heating time with water cooling on size distribution of grinding product (6 kW, 500 g).

Size /mm	Yield /%					
	0s	10s	20s	40s	60s	80s
-3.2+2	3.24	3.61	2.96	2.44	1.84	2.24
-2+1	20.36	21.17	21.94	16.88	12.94	11.58
-1+0.425	35.04	34.41	34.01	33.72	34.06	35.33
-0.425+0.15	17.22	17.05	16.69	18.78	20.79	20.91
-0.15+0.074	5.66	5.68	5.82	6.58	7.84	7.40
-0.074+0.038	4.59	4.33	4.50	5.42	5.71	5.77
-0.038	13.88	13.74	14.08	16.18	16.81	16.77