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Microwave absorption characteristics of anthracite during pyrolysis

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ABSTRACT

The microwave absorption characteristics of a Korean anthracite were explored by determining its dielectric properties (relative dielectric constant and loss factor) using the cavity perturbation technique up to approximately 1000 °C at 915 MHz and 2450 MHz in ultra-high purity argon. The dielectric parameters increase gradually below 800 °C, showing an interestingly linearly increasing tendency with temperature essentially associated with breaking of chemical bonds and removal of volatiles during pyrolysis. With a further increase in temperature, both parameters increase considerably mainly because of enhanced electronic conduction caused by more free charges induced and potential interfacial polarization as a result of build-up of charges at contact areas or interfaces. The continuously decreasing microwave penetration depth within the temperature range further reveals the increased microwave absorption in the coal during pyrolysis. According to the reflection loss patterns of the coal, the maximum microwave absorption is achievable at an appropriate sample thickness. This corresponds to the minimum reflection losses of −44.18 dB and −13.03 dB for sample thicknesses of 0.02 m and 0.04 m at respectively 915 MHz and 2450 MHz. The sample thickness substantially influences the microwave absorption in coal during pyrolysis, especially in the initial stage.

1. Introduction

Use of microwave energy for efficient coal unitization has gained broad attention due to the important and stimulating characteristics of microwave irradiation, such as selective and volumetric heating [1,2]. The representative applications of microwave in this area include coal drying [3], grinding [4], beneficiation [5,6], coking [7], liquefaction [8,9], etc. As an important technique for coking, liquefaction and related fuel conversion uses, microwave coal pyrolysis has been extensively explored [10]. It was reported that the application of microwave to coal pyrolysis may substantially reduce reaction time and temperature, and the pyrolysis performance is dictated by the microwave absorption capability of coal which varies with its dielectric properties [11]. In fact, the dielectric properties of coal depend on a variety of factors, including temperature, microwave frequency and coal composition [12]. Early studies explored the dielectric properties of coal at low temperature [13–15]. It was demonstrated that the organic fraction of coal has poor dielectric properties and thus weak microwave response at room temperature. Conversely, some components in the coal matrix, such as moisture and sulfur-bearing minerals, e.g., pyrite, have much higher dielectric loss that determines the microwave absorption in coal [16]. When temperature is elevated, considerable variation of the dielectric properties of coal may occur due to structure transformation during pyrolysis along with removal of moisture and volatiles [17]. This usually causes a sudden change in its microwave absorption capability that eventually impacts on the efficiency of microwave coal pyrolysis.

By now, most previous studies focused on a limited temperature range, usually below 200 °C, and few considered the microwave dielectric properties and microwave absorption in coal within a large temperature range at allowed industrial frequencies [17,18]. With growing applications of microwave irradiation in the pyrolysis of coal, there is increasing demand for detailed dielectric characterization and microwave absorption capability of coal during pyrolysis for improving microwave coal pyrolysis and other related microwave coal processing technologies [19]. The objective of this study is to explore the microwave absorption characteristics of an anthracite during pyrolysis by evaluating its microwave penetration depth and reflection loss on the basis of dielectric properties measurements in the temperature range up to about 1000 °C at 915 MHz and 2450 MHz (the most commonly used frequencies). The findings may help to reveal the mechanism of microwave loss in anthracite and promote relevant microwave pyrolysis processes.

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2. Experimental

2.1. Coal sample characterization and preparation

In the present study a Korean anthracite was employed for dielectric properties measurements. The proximate and ultimate analyses of the sample in Table 1 show that the coal has low inherent moisture and high ash contents. For dielectric characterization, the sample was ground into powder with particle size smaller than 75 μm. After drying at 105 °C for 15 h, the powder was unidirectionally pressed in a tungsten carbide (WC)-lined die at about 180 MPa to prepare 3 pellets having a diameter of 3.67 mm and a total piled length (height) of 13.62 mm. The initial pellet apparent density was 1.69 g cm^{-3}, and the final density at the end of the experiment was 1.62 g cm^{-3}, accompanying 7% mass loss. The pellets with small diameter were prepared to minimize the interference of air on the dielectric measurements which require homogeneous microwave distribution within the sample to ensure measurement accuracy [20].

2.2. Dielectric properties measurements

As aforementioned, the dielectric response of a substance varies with temperature and microwave frequency. It is usually expressed as complex permittivity (ε) shown by [21,22]

$$\epsilon = \epsilon_0 \epsilon_r = \epsilon_0 \left(\epsilon_r^{'} - j \epsilon_r^{''} \right) \tag{1}$$

where ε_0 is the free-space permittivity (8.854×10^{-12} F m^{-1}); ε_r is the complex relative permittivity, and j is the imaginary unit. In practice, the complex relative permittivity is used to quantify the dielectric response for convenience because of the small absolute values of permittivity for most materials. It can be observed that the complex relative permittivity consists of two parts: relative dielectric constant ε_r' and relative dielectric loss factor ε_r''. Specifically, ε_r' measures the ability of the dielectric material to store electrical energy, whereas ε_r'' quantifies the loss of electrical energy in the material. Both parameters impact on microwave absorption in materials and thus the efficiency of microwave processing [23,24].

The dielectric properties (ε_r' and ε_r'') of the coal during pyrolysis were determined at 915 MHz and 2450 MHz using the cavity perturbation technique in the present study. This technique was based on the measurement of the differences in the microwave cavity response caused by insertion of sample, namely the frequency shift and change of quality factors between a cavity with an empty sample-holder and the same cavity with a sample-holder plus the sample, used for calculation of the complex relative permittivity. The measurement system mainly consisted of a resistance furnace that heated sample to the designated temperature and a cylindrical TM_{0n0} resonant mode cavity (ø580 mm × 50 mm) in which the aforementioned dielectric response differences caused by sample were detected and recorded in a Hewlett Packard 8753B vector network analyzer. According to the frequency shift and change in quality factor measured, the complex electric susceptibility (χ_e) was computed and used for calculation of dielectric parameters. The detailed descriptions of the cavity perturbation technique and major apparatus can be found in the literature [17,25].

For determination of dielectric properties of the coal during pyrolysis, each measurement was completed by inserting a thin (approximately 0.7 mm in diameter) low dielectric loss quartz disc flat onto the bottom of a "flow-through" holder. The coal sample was step-heated to about 1000 °C from room temperature (21 °C) and heating in 50 °C steps with a ramp rate of 5 °C min^{-1} in 0.005 L min^{-1} flowing ultrahigh-purity argon for prevention of oxidation and combustion. The entire measurement process was pre-programmed and controlled by the LabVIEW software.

2.3. Dielectric loss tangent

Dielectric loss tangent ($tan\delta$) is a parameter that evaluates variations of dielectric constant and loss factor simultaneously. It is defined as the ratio between relative dielectric loss factor and relative dielectric constant, given as

$$\tan\delta = \frac{\epsilon_r''}{\epsilon_r'} \tag{2}$$

In general, the larger the loss tangent, the greater the attenuation/microwave absorption which converts to heat under microwave irradiation as the wave travels through the material.

2.4. Penetration depth

Microwave penetration depth (D_p) is an vital parameter in evaluating the microwave absorption capability of materials and in proper microwave cavity design. It is defined as the depth within the material where the onward traveling microwave power declines to 1/e of its original value at the material surface [26]. With the dielectric parameters measured, D_p of the coal is determined by the equation as follows [26,27]:

$$D_p = \frac{\lambda_0}{2\pi (2\epsilon_r')^{1/2}} \left\{ \left[1 + \left(\frac{\epsilon_r''}{\epsilon_r'} \right)^2 \right]^{1/2} - 1 \right\}^{-1/2} \tag{3}$$

where λ_0 is the free-space microwave wavelength.

2.5. Reflection loss

In view of the impact of sample-loaded metallic cavity on microwave absorption in materials, reflection loss (RL) can be used to further evaluate microwave absorption capability of the coal [17]. For a coal slab backed by a metallic cavity wall (a perfect electric conductor), the vertically incident microwaves of unit amplitude usually induce reflected waves that transmit over the reverse path. In accordance with the transmission-line theory, RL of the coal slab is derived as

$$RL = 20 \log \left| \frac{\sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh\left(j \frac{2\pi f}{c} \sqrt{\mu_r \varepsilon_r} d \right) - 1}{\sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh\left(j \frac{2\pi f}{c} \sqrt{\mu_r \varepsilon_r} d \right) + 1} \right| \tag{4}$$

where μ_r is the complex relative permeability assumed to be 1 in the present study; c is the free-space microwave velocity; and d is the thickness of the coal slab.

Table 1
Proximate and ultimate analyses of anthracite.

Proximate analysis (air dried)					Ultimate analysis (dry, ash-free basis)				
Parameter	Inherent moisture	Volatile matter	Fixed carbon	Ash	C	H	N	S	O[a]
Value (wt.%)	1.39	2.44	69.59	26.58	92.71	2.72	1.01	0.43	3.13

[a] by difference.