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Dielectric Properties and Temperature Increase of Zinc Oxide Dust Derived from Volatilization in Rotary Kilns

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ABSTRACT

The present study measures dielectric properties (ε', ε'' and $tan\delta$) of zinc oxide dust produced in a rotary kiln, using a cavity perturbation method with microwaves at 2.45 GHz. The effect of apparent density of the dust on the dielectric properties was determined. The results indicated that zinc oxide dust has excellent absorbing performance and can reach 800 ºC in 6 minutes. The apparent density of zinc oxide dust and the microwave penetration depth were also related.

KEYWORDS: Zinc oxide dust, dielectric properties, apparent density, thermal behavior.

INTRODUCTION

Zinc is an important metal used in the metallurgy, chemical and textile industries. It is mostly extracted from sulfide ore, and by recycling of zinc wastes, such as zinc ashes, dross, flue dusts, sludge and residues of zinc generated in various chemical and metallurgical processes [Jha et al., 2001; Dutra et al., 2006; Asadi Zeydabadi, et al., 1997]. The raw materials contain several impurities such as lead, cadmium, chromium, nickel, other non-ferrous metals, and even small amounts of indium [Xie 2008; Fan et al., 2011; Zhu, 2007; Zhu et al., 2006; Bruckard et al., 2005], while other impurities, such as fluorine and chlorine, come from different secondaries sources. Nowadays, more than 30% of the worldwide zinc supply comes from recycled zinc, with a high content of fluorine and chlorine that strongly affects the zinc electrolytic process. In this process, concentration of fluorine must be less than 80 mg/L, and chlorine less than 100 mg/L [Güresin et al., 1998, Şahin et al., 2000].

At the present stage, the removal of fluorine and chlorine is conducted by two ways, pretreatment removal, and removal by leach liquor. In the case of high fluoride or chloride content materials, multiple hearth furnace, rotary kiln and other roasting approaches are used; the removal efficiency is low. Using caustic washing to take off the fluorine and chlorine will lead to difficult control of waste water, so it is necessary to explore a new method for removal of these elements [Mason et al., 2006; Bodson 1977; Miao et al., 2012].

Microwave metallurgy as a kind of high efficiency, clean green technology has been developing rapidly [Xia et al., 2000]. In general, microwave heating is unique and offers a number of advantages over conventional heating such as: (1) Selective, high rapid and efficiency heating; (2) Catalyze chemical reaction, lower reaction temperature, reaction time and consumption, save energy; (3) Does not produce any gases itself, being one of the effective ways to perform clean processing; (4) Easy Control, Making materials obtain / lose heat source instantly. Successively appeared some new technology on microwave drying, microwave assisted grinding, microwave assisted reduction, microwave strengthening leaching [Metaxas et al., 1983; Clark et al., 2000; Salsman et al., 1996; Jones et al., 2007; Al-Harahsheh et al., 2004].

From zinc oxide dust, chloride sulfide exhibits strong wave absorption, however the zinc oxide is a weak microwave absorber [Peng, et al., 1997; Zhang, et al., 2011]. Because of microwave selective heating, it can strengthen the impurities separation of volatile components, helping to achieve the goal of removing fluorine and chlorine. 300 g of zinc oxide dust containing 0.28% of Cl was used in the microwave roasting dechlorination experiments. Under a power of 1.2 kW at 2450 MHz, The roasting temperature was constant at 650 °C for 30 min. During the roasting process, the samples were stirred fully while air was extracted from the cavity. After microwave treatment, chlorine content can be reduced to 0.012% and removal rate was 85% [Peng et al., 2011]. The results indicate that microwaves can be used for treatment of zinc oxide dust.

Microwave heating depends the dielectric loss of the material, this important parameter quantify the interaction between microwaves and materials [Lin 1979; Dong 1991]. Permittivity (or dielectric properties) of materials vary with chemical composition, as the changes during reaction, and temperature, which is also changing during microwave heating; in turn, distribution of the electromagnetic field and absorbed power is affected. In order to determine the complex permittivity of the material as function of temperature aimed to optimize the design of a microwave reactor for metallurgical processes, numerical methods were used to mimic the distribution of the electric field and temperature field in microwave cavity.

Dielectric properties are defined in terms of complex permittivity (ε), composed of a real part (ε' dielectric constant) and an imaginary part (ε'' dielectric loss factor) by the equation: $\varepsilon = \varepsilon' - j\,\varepsilon''$ [Nelson et al., 2012].

Loss tangent ($tan\delta$), is a parameter used to describe how well a material absorbs microwave energy, is the ratio of dielectric loss factor and the dielectric constant ($tan\delta = \varepsilon''/\varepsilon'$) [Patil et al., 2012]. A material with a higher loss tangent will heat faster under a microwave field as compared to a material with a lower loss tangent. Dielectric constant and loss tangent are functions of frequency, material homogeneity and anisotropy, moisture, and temperature of the material. Various measurement techniques are available for the experimental determination of dielectric properties.

In order to describe microwave roasting of zinc oxide dust for removing fluorine and chlorine, the dielectric properties (ε', ε'' and $tan\delta$) of zinc oxide dust were determined using microwave cavity method. The thermal behavior of the materials was also evaluated during the microwave heating of the materials. The idea was to find elements for expanding possibilities of applications in metallurgy.

EXPERIMENTAL

Materials

The zinc oxide dust was obtained from Lead and Zinc Smelting Enterprises Company, Yunnan Province, P. R. China. Zinc oxide dust was dried at 85 ºC, then the chemical composition of raw materials was

Table I. Chemical compositions of zinc oxide dust sample (mass fraction, %)

Component	Zn	Pb	Fe	K	S
Content (%)	50.10	10.63	3.45	2.92	2.21
Component	Cl	Bi	Ca	F	In
Content (%)	1.30	0.91	0.61	0.43	0.14

determined by means of XRD. The chemical composition of the zinc oxide dust is listed in Table I. The XRD spectra of the zinc oxide dust samples are shown in Figure 1. It can be seen from this figure that ZnO is the main phase of zinc oxide dust, lead is mostly as PbOHCl and $K_2Pb(SO_4)_2$, in addition, there is also present a small amount of Fe_2O_3.

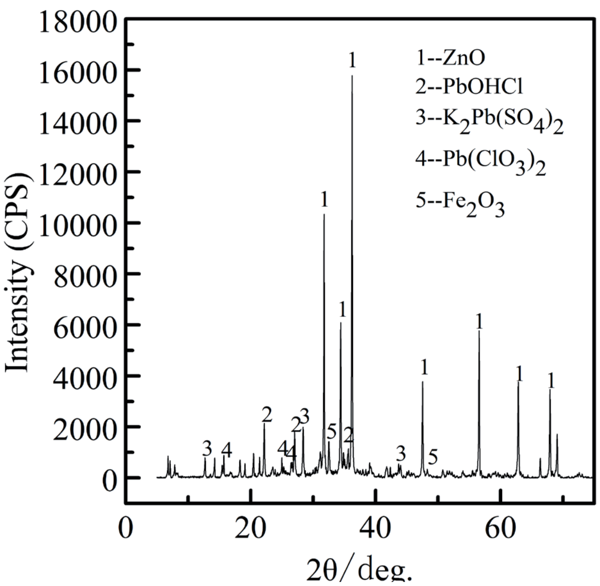

Figure1. XRD patterns of zinc oxide dust.

The results of Table I and Figure 1 show that zinc oxide dust has a high content of lead and zinc, and the content of fluorine and chlorine element, which do harm for the zinc electrolysis process certain combined lead and zinc into fluorine and chlorine compounds. Fluorine and chlorine were bound to enter the leaching solution in the leaching process and caused excessive leach problems.

Test device for temperature measurement in microwave field

The main experiment equipment was a 3 kW box-type of microwave reactor, which was designed by Key Laboratory of Unconventional Metallurgy, Ministry of Education, Kunming University of Science and Technology P. R. China.

The experimental device (Figure 2) contains automatic temperature control, microwave heating frequency of 2450 MHz, power up to 3kW, continuously adjustable, the thermocouple was shielded and able to measure the temperature in range from 0 to 1300 °C. The

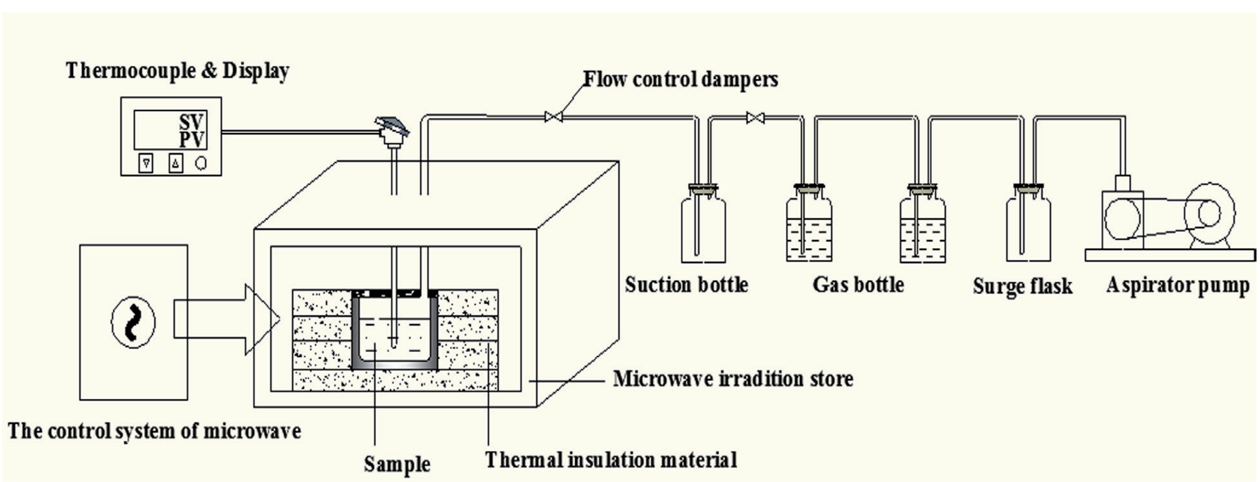

Figure 2. The diagram of microwave temperature characteristics measuring device.

material supporting body was a mullite crucible with good wave-transparency and heat shock properties. The inner diameter is 90 mm and the height is 120 mm, The smoke soot absorption system was composed by the dust collection bottle, two water bottles, an alkali absorption bottle, a buffer bottle and a miniature pump. It can collect and absorb the flue dust that was generated in the experimental process.

Resonant cavity perturbation method

The resonant cavity perturbation method [Huang et al., 2005] is widely used because of its high accuracy. The method can be expressed as follows [Carter, 2001],

$$\frac{\Delta\omega}{\omega} = -\omega_0(\varepsilon_r^{'} - 1)\int_{V_e} E_0^* \cdot E dv / (4W) \quad (1)$$

$$\frac{1}{Q} - \frac{1}{Q_0} = 2\varepsilon_0 \varepsilon_r^* \int_{V_e} E_0^* \cdot E dv / (4W) \quad (2)$$

$$W = \int_V [(E_0^* \cdot D_0 + H_0^* \cdot B_0) + (E_0^* \cdot D_1 + H_0^* \cdot B_1)] dv \quad (3)$$

where $\Delta\omega = \omega - \omega_0$, $\Delta\omega$ is the frequency deviation; ω_0 is the resonance frequency of cavity in the unperturbed condition; ω is the corresponding parameters of the cavity loaded with the sample; ε' and ε'' are the real and the imaginary part of the complex permittivity of the sample, respectively; V and V_e are the volumes of the cavity and the sample, respectively; E_0^*, D_0, H_0^*, and B_0 are the fields in the interior of the sample; E is the field strength of cavity; dv is the elemental volume; Q_0 is the resonance frequency of cavity in the unperturbed condition, respectively; Q is the corresponding parameters of the cavity loaded with the sample; W is the storage energy; D_l and B_1 are the added value of the electric displacement and magnetic induction intensity respectively.

An scheme of the cavity perturbation is shown in Figure 3, the zinc oxide dust samples were put in the microwave cavity resonator sensor.

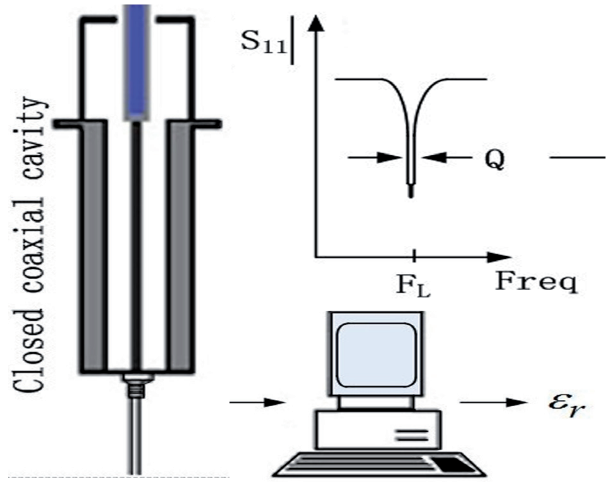

Figure 3. Scheme of the cavity perturbation.

Test equipment and testing process

Dielectric parameters measuring device scheme are shown in Figure 4. The testing equipment is a German Puschner company dielectric parameter tester (Dielectric kit for Vials). The device consists of a microwave power source, a directional coupler, a microwave receiver and cavity resonator. The microwave signal receiver of AD-8320 integrated circuit can detect the signal amplitude and phase. The resonator was used to hold in the analyze cavity. The test control unit is via a USB data cable connected to the computer that calculates the dielectric parameters. The test sample was placed in a

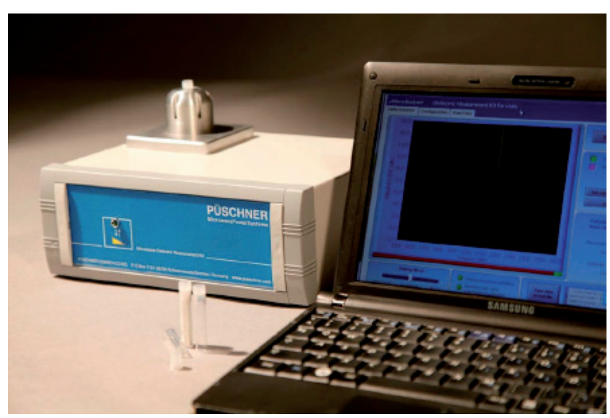

Figure 4. Zinc oxide dust dielectric constant measurement device scheme.

small bottle and the vials were taken into the cavity resonator through the opening holes. By comparing the resonance frequency and factors before and after, according to the test cavity perturbation theory, the dielectric parameters were calculated.

The dried zinc oxide dust was divided into 11 portions that were weighted and placed in sealed quartz standard tubes of known volume at 21 ± 1 °C for calculating their density. The system error of the dielectric coefficient real parts was estimated to be 3 to 5% [Dong, 1991] during the perturbation method tests, deionized water (the real part of permittivity is 78 F/m) was used and polytetrafluoroethylene (the real part of permittivity is 2.08 F/m) as standards, resulting in 76.79 F/m and 2.04 F/m, respectively, 1.55% and 1.92% differences. These results illustrated that the measurement of small volume perturbation was feasible by using this method.

RESULTS AND DISCUSSION

Dielectric property test results of zinc oxide dust and analysis

The measured dielectric properties are related to many factors, such as physical properties, density, humidity and frequency.

Dielectric Properties Measuring Process: first, put the sample of zinc oxide dust into the air-tight quartz glass standard tube; Second, measure the weight of standard tube before and after the sample in to determine the sample mass; Third, calculate the apparent density, and measure the dielectric properties through putting the standard tube carried with sample into the resonant cavity.

Apparent density affecting dielectric properties

The effect of apparent density on the dielectric constant, dielectric loss and loss tangent, respectively are shown in Figures 5 and 6.

Figures 5 and 6 show that the relation of the value of ε' and ε'' at 2.45 GHz to the different apparent density for zinc oxide dust. The relationship found is in good agreement with the values reported by Hotta et al. [2009].

The relation between the values (ε') and the apparent densities (ρ) can be expressed by the following equation,

$$\varepsilon' = 1.6664\rho^2 - 6.8424\rho + 8.4861 \quad (R^2 = 0.99545). \tag{4}$$

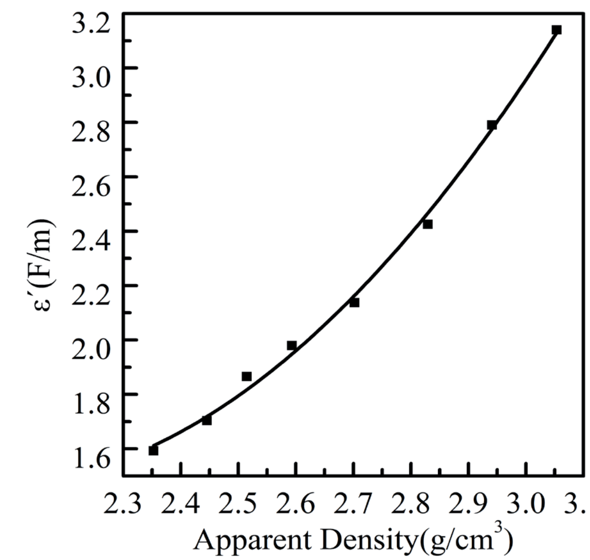

Figure 5. Effect of apparent density on dielectric constant.

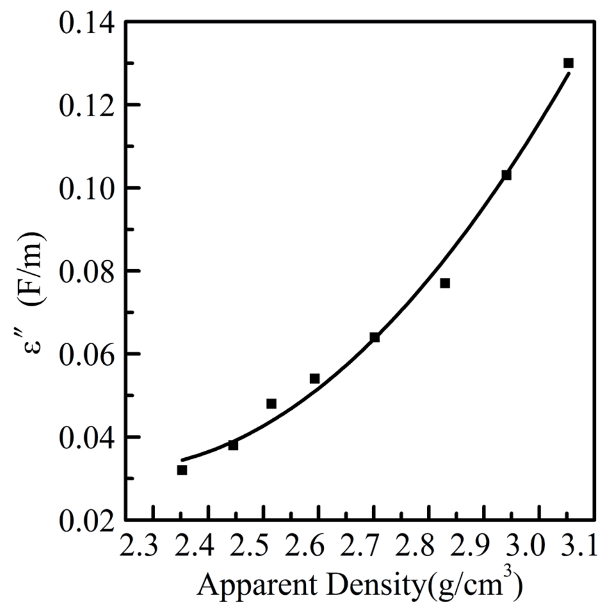

Figure 6. Effect of apparent density on loss factor.

The relation between the values (ε'') and the apparent densities (ρ) can be expressed by the following equation,

$$\varepsilon'' = 0.1389\rho^2 - 0.61807\rho + 0.71976 \quad (R^2=0.98689). \tag{5}$$

Figure 7 shows that value of $tan\delta$ does not change significantly with increasing apparent density at first, and then the value of $tan\delta$ varies significantly. Because of the increase in apparent density narrow the material particles gap and in the microwave penetration process, it reduces the impact on the determination of air and improves the capacity of microwave absorption.

Apparent density effect on microwave penetration depth

The microwave penetration depth is known as the distance from the surface to the inside of the material, where the microwave field intensity is reduced to $1/\varepsilon$ of the original field intensity. The expression reported elsewhere [Guo et al., 2010; Kumar et al., 2007; Peng et al., 2010]:

The size of the sample determines if uniform heating is posible [Peng and Yang, 1997].

$$D_P = \frac{\lambda_0}{2\sqrt{2}\pi\sqrt{\varepsilon'[\sqrt{1+(\frac{\varepsilon''}{\varepsilon'})^2}-1]}} \tag{6}$$

D_P determines the material microwave heat uniformity at 2.45 GHz, λ_0=12.24 cm. The results showed that the penetration depth (D_P) decreased when the apparent density increased. The values found are shown in Figure 8, and the regression equation is shown below:

$$Dp = 68.08331\rho^2 - 408.9273\rho + 688.59395 \quad (R^2 = 0.98018). \tag{7}$$

Temperature rising of zinc oxide dust

Temperature increase Measuring Process: first, dried and grinded sample was put in mullite crucible with heat preservation material around and all of them were carried in microwave reactor. Second, turn on the power of microwave system and off-gas absorption system, adjust the instrument parameters. Third, record the trend data of the temperature increased with time every 20 seconds, and when the data kept the same, the whole experiment ended.

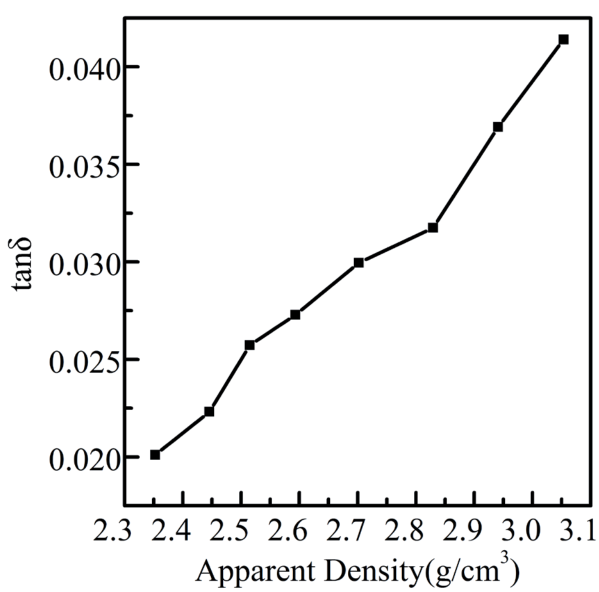

Figure 7. Effect of apparent density on loss tangent.

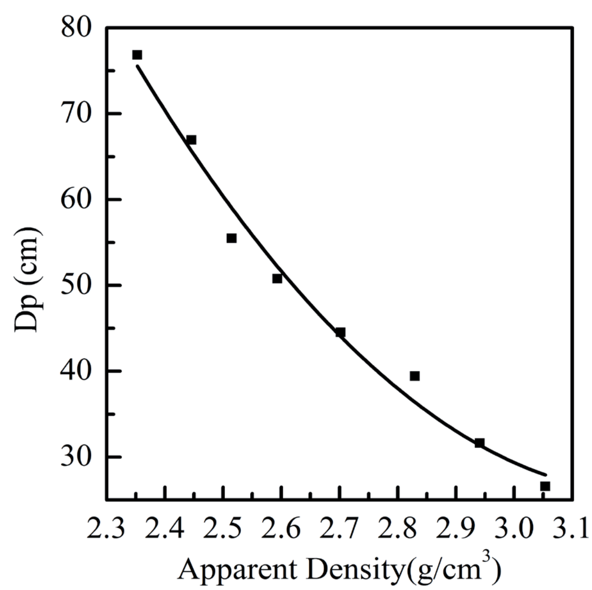

Figure 8. Apparent density effect on microwave penetration depth.

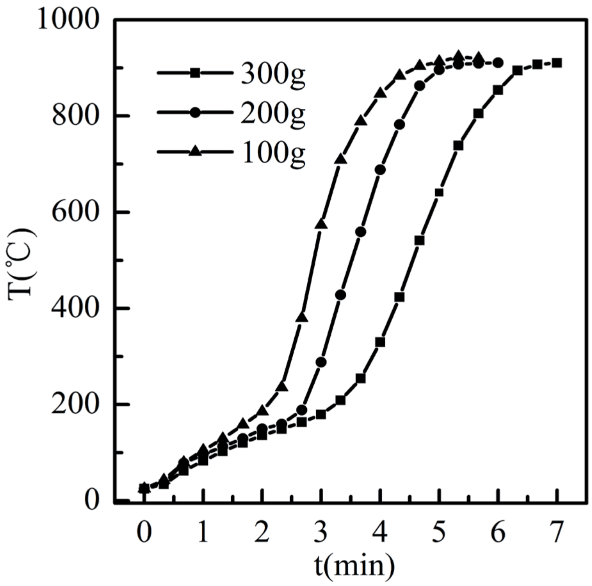

Figure 9. Zinc oxide dust heating by microwaves at 900 W.

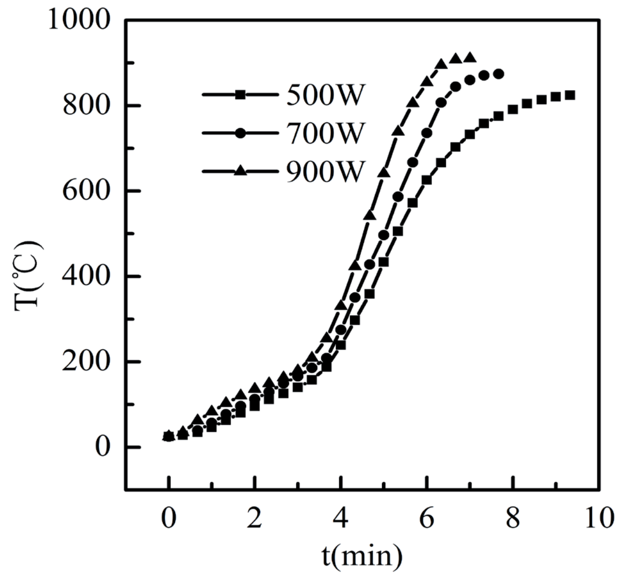

Figure 10. Zinc oxide dust heating rate (300 g) curve at different microwave power.

The temperature-rising and the amount of material in the microwave field are closely related, as shown in Figure 9, under microwave power is 900 W. The average heating rate were 142.01 °C/min, 180.69 °C/min, and 211.5 °C/min, for 300 g, 200 g, and 100 g respectively. The relation reported by Chen et al. [2007], the greater the dust mass, the lower the heating rate, which is consistent with the experimental results.

Microwave power affecting temperature rising

The heating curve of 300 g of zinc oxide dust exposed to 500 W, 700 W, and 900 W microwaves is shown in Figure 10. The experiment result is similar with test conclusions for microwave heating low-grade nickel oxide ore [Hua et al., 2000].

Dechlorination of zinc oxide dust

The comprehensive approach of exploiting zinc oxide dust provides an effective way to alleviate the pressure on the shortage of raw materials in the lead and zinc industry. However, the high energy consumption for processing the dust with conventional methods due to the high content of chlorine is the primary obstacle to this exploitation. This paper proposes to investigate the new technology of microwave heating for removing chlorine from the zinc oxide dust with high contents of chlorine by taking full advantages of microwave radiation of its unique manner of energy transmission and selective heating feature and making use of the property of halide of strong microwave absorption. Dielectric properties and temperature rise of The zinc oxide dust mixture were investigated. According to the volatile characteristics of lead and zinc chloride at high temperatures [Li et al., 2010, Li et al., 2009, Zhang et al., 2011, Wang et al., 2007], some exploratory experiment researches of dechlorination from zinc oxide dust by microwave were developed adopting the experiments in figure 2.

A near 90% dechlorination of zinc oxide dust could be achieved which would satisfy the requirements of the wet smelting electrolysis process. 300 g of zinc oxide dust is dechlorinated at 900 W. The optimal process parameters were identified to be a stirring speed of 60 rpm, a roasting temperature of 650 °C, and a roasting duration of 60 min. These conditions meet the Cl content required by leached solutions in the production of electrolytic zinc. The results obtained in the present laboratory study showed that high Cl zinc oxide dust could be treated effectively by microwave roasting.

The successful outcome of this research will provide the theoretical basis for effectively separating fluorine and chlorine as well as efficiently producing zinc with low energy consumption from the zinc oxide dust.

CONCLUSION

The complex permittivity and the temperature rising of zinc oxide dust from rotary kilns were measured.

An expression for relating the dielectric properties and penetration depth during the heating of zinc oxide dust presented.

Zinc oxide dust is a good microwave absorber and can be heated quickly in the microwave field.

Thermodynamic conditions for removing fluorine and chlorine by roasting at 800 °C were achieved in 6 minutes with this particular experimental arrangement.

ACKNOWLEDGEMENTS

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