

Complex Permittivity and Permeability of SiO₂ and Fe₃O₄ Powders in Microwave Frequency Range between 0.2 and 13.5 GHz

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The real and imaginary parts of relative complex permittivity (ϵ' and ϵ'') and relative complex permeability (μ' and μ'') were measured for powder and bulk SiO₂ and Fe₃O₄ in the frequency range of 200 MHz–13.5 GHz. The ϵ' and ϵ'' values of SiO₂ powder samples are in good agreement with the previous data. The ϵ' values of powder SiO₂ and Fe₃O₄ samples with the relative density below 1 are smaller than the values estimated using the linear relation between ϵ' and the relative density, and larger than those estimated using the Lichtenecker's logarithm mixed law. The μ'' values of powder Fe₃O₄ samples show the peaks in the frequency range of 706 MHz to 3.21 GHz. The frequency at the peak of the μ'' vs. frequency curve decreases with an increase in the particle size of Fe₃O₄ sample. All the powder samples with an identical relative density have almost the same μ'' values around 2 to 3 GHz irrespective of the particle size. Above ca. 3 GHz, the μ'' values of all samples become smaller as the measurement frequencies become higher. These results suggest that 2.45 GHz, the frequency of the most commercially prevailing microwave generators, may be the most suitable one for microwave heating of iron ores.

KEY WORDS: permittivity; permeability; Fe₃O₄; SiO₂; relative density; particle size; microwave frequency.

1. Introduction

Nowadays, CO₂ emission reduction and energy conservation are urgent tasks owing to the initiation of Kyoto Protocol adopted for the prevention of global warming. Since in the year 2007, as much as 11% of the CO₂ emission is attributed to the blast furnace-type iron making in Japan, new iron making process should be invented based on innovative theories and mechanisms. So far, ca. 500 kg of coke is consumed to produce 1 ton of pig iron. Consumption amount of coke for the reduction of iron ore and carburization is ca. 225 kg, and the residual 275 kg is used to heat the raw materials and cause the reduction reaction which is an endothermic reaction. If heat source for the latter can be supplied by electric energy produced by no CO₂ emitting electric sources such as nuclear power, wind power and solar power, CO₂ emission could be dramatically reduced. We have focused on the microwave heating as a new heating source for iron making.¹⁻⁴⁾

Ishizaki *et al.*²⁾ have produced the pig iron from the carbon composite iron ore pellet composed of the mixture of magnetite ore (Romeral) and coal (Robe River) powders by means of microwave heating. The output power of the microwave generator was 3 kW at 2.45 GHz. The temperature reached 1350°C ca. 500 s after the onset of the heating, and subsequently the power was switched off. It has been found that the impurity concentrations in the obtained pig iron are lower than those of pig iron produced by blast furnace. This

study implies that iron making from powder raw materials using microwave heating could yield the low temperature operation and the highly-pure pig iron production: The production of highly-pure pig iron is considered to be relevant to the rapid heating of raw materials.⁵⁾

In order to scale up this process to practical use, however, we have to solve several problems. Some of them are as follows; (i) development of refractories with poor microwave absorptivity, (ii) searching for the suitable dimensions and shapes of raw materials with good microwave absorptivity, and (iii) searching for an appropriate microwave frequency for optimal operation. These all should be solved to improve the energy efficiency, and to solve these problems, the microwave-material interaction should be elucidated.

Microwaves are electromagnetic waves, the frequency range of which is from 0.3 to 300 GHz (corresponding to wavelengths ranging from 1 mm to 1 m). The interactions between a material and electromagnetic fields can be described by the following equation.

$$\mathbf{D} = \epsilon \mathbf{E} = \epsilon_0 (\epsilon' - j\epsilon'') \mathbf{E} \dots \dots \dots (1)$$

$$\mathbf{B} = \mu \mathbf{H} = \mu_0 (\mu' - j\mu'') \mathbf{H} \dots \dots \dots (2)$$

Where \mathbf{D} electric flux density, \mathbf{B} magnetic flux density, \mathbf{E} electric field strength vector, \mathbf{H} magnetic field strength vector, ϵ_0 permittivity of vacuum, μ_0 permeability of vacuum, $\epsilon = \epsilon' - j\epsilon''$ relative complex permittivity of material,

$\mu = \mu' - j\mu''$ relative complex permeability of material. Microwave absorption can be described by the following equation.

$$P = \frac{1}{2} \sigma |E|^2 + \pi f \epsilon_0 \epsilon'' |E|^2 + \pi f \mu_0 \mu'' |H|^2 \dots\dots\dots (3)$$

Where $|E|$ electric field amplitude, $|H|$ magnetic field amplitude, σ electrical conductivity, f microwave frequency, ϵ'' imaginary part of relative permittivity, μ'' imaginary part of relative permeability. The first, the second and the third terms of the right hand side of Eq. (3) correspond to the microwave absorptions due to the Joule loss, the dielectric loss and the magnetic loss, respectively. Morita *et al.*⁶⁾ have measured the heating rate of blast furnace slag using a microwave furnace and have reported that it becomes faster as the larger amount of Fe_3O_4 phase is included in the slag. They have concluded that the blast furnace slag is heated by the dielectric loss due to Fe_3O_4 . On the other hand, Cheng *et al.*⁷⁾ have measured the heating rates of some metal, carbide and oxide powders at the H (magnetic) anti-node (where the E field is nearly zero) or the E (electric) anti-node (where the H field is nearly zero) using a 2.45 GHz single mode cavity, and have found that electro-conductive powders including Fe_3O_4 are more efficiently heated in the magnetic field. They have insisted that more detailed consideration for the effects of the magnetic field on materials are strongly required. Ishizaki *et al.*²⁾ have heated a carbon composite iron ore pellet composed of the mixture of magnetite and coal powders using a 2.45 GHz multi mode microwave furnace, and have reported that the temperature rapidly increases up to *ca.* 600°C and then levels off for a while. They have suggested that a carbon composite iron ore pellet is initially heated by the magnetic loss of Fe_3O_4 and that the stagnation of the temperature increase is caused by the disappearance of magnetization above the Curie temperature (585°C).⁸⁾ Since the magnetic loss is relevant to the imaginary part of the complex permeability, it is necessary to investigate the complex permeability of Fe_3O_4 powders in order to search for the optimal condition regarding the microwave irradiation on raw materials of ironmaking.

The aim of this study is to measure the complex permeability of Fe_3O_4 powders as a function of the powder size and the microwave frequency by means of the coaxial transmission line method. This method enables us to carry out the simultaneous measurement of complex permittivity and permeability. In order to confirm the feasibility of the present measurement, the complex permittivity of SiO_2 was also measured so as to compare the results with the previous values.

2. Experimental

2.1. Sample Preparation

A fused silica powder (99.5% purity) with the average particle size of *ca.* 30 μm and a fused silica bulk having the impurity of 5 ppb sodium were used for the permittivity and permeability measurements. The bulk sample was machined into a thick tube (3.04 mm in inner diameter, 7.00 mm in outer diameter and 3.57 mm in height) with a grinder and a wheel dresser to be fitted to the coaxial sam-

ple holder. As for Fe_3O_4 powders, four samples were used; three were spherical powders (99.9% purity) having different particle sizes of (i) 100–180 μm , (ii) 38–62 μm , (iii) 50–60 nm and one was an acicular powder (98% purity) with the particle size of 0.1 \times 0.8 μm . Spherical Fe_3O_4 powder samples were achieved by means of the mesh control of Fe_3O_4 powders having the particle size less than 180 μm . Particle sizes of Fe_3O_4 powders were confirmed by scanning electron microscope (SEM) observation. A sintered and a single crystal Fe_3O_4 bulk were also used. The bulk samples were made in the following manner. Fe (99.9% purity) and Fe_2O_3 (99.99% purity) powders were mixed to make the atomic ratio of Fe/O be 2.98/4 and formed into a rod. The sintered sample was prepared by sintering the rod at 1373 K for *ca.* 16 h in vacuum and then quenching it to room temperature. The single crystal was grown in a flow of argon gas (99.99% purity) from the sintered rod by the floating zone method. To avoid the sample from oxidation, oxygen was removed from the argon gas by passing it through sponge zirconium at *ca.* 1073 K. Those samples were machined with a grinder and a wheel dresser so as to be fitted to the coaxial sample holder (Sintered and single crystal samples were 4.86 and 3.66 mm in height, respectively). It was confirmed by X-ray diffraction analysis (XRD) that the phases of all Fe_3O_4 samples were only composed of Fe_3O_4 before and after the measurement. Prior to the measurement, both SiO_2 and Fe_3O_4 samples were dried at elevated temperatures to get rid of the adsorbed moisture.

2.2. Permittivity and Permeability Measurement

The real and imaginary parts of permittivity (ϵ' and ϵ'') and permeability (μ' and μ'') were measured by the coaxial transmission line method using the network analyzer (Agilent Technologies N5230A), a coaxial cable and a 7 mm coaxial sample holder (KEAD CSH2-APC7) over the microwave frequency range between 0.2 and 13.5 GHz. After measuring the transmission and reflection (S-parameter) of the irradiated microwave, the permittivity and permeability were calculated by the algorithm of Nicolson–Ross model. When measuring a powder sample, the sample was sandwiched between two teflon tubes with *ca.* 3 mm in height as shown in Fig. 1. Since the permittivity of teflon is larger than that of air ($\epsilon' \approx 2.1$ at 298 K), its influence on the permittivity and permeability measurement of the powder samples is not negligible. Therefore, the permittivity and permeability of teflon were measured individually, and the influences were computationally eliminated from the apparent

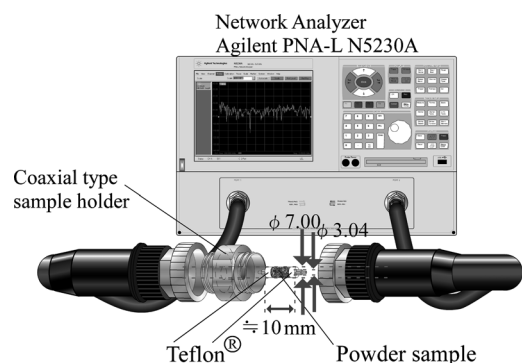


Fig. 1. Schematic illustration of the experimental apparatus.

permittivity and permeability of the powder samples to obtain the real values. This measurement technique has been described in elsewhere.⁹⁾ Only the powder sample with the particle size of 50–60 nm was measured in the argon gas atmosphere because the oxidation rate of nanoparticle is fast. In order to confirm the feasibility of the computer program, the ϵ' value of air was measured using the teflon tubes. As a result, the obtained values of ϵ' were between 1 and 1.3. The apparent density of a powder sample was calculated by the sample length and weight.

3. Results

3.1. Permittivity and Permeability of SiO₂

Figures 2(a) through 2(d) show the frequency dependency of (a) the real parts of permittivity ϵ' , (b) the imaginary parts of permittivity ϵ'' , (c) the real parts of permeability μ' , and (d) the imaginary parts of permeability μ'' on the powder SiO₂ samples having the relative densities of 46.8–70.2% and a bulk SiO₂ sample. A powder SiO₂ sample with each relative density was measured only once. The bulk sample was measured five times, and the average values and the standard deviations are plotted in Fig. 2. The relative density of a powder sample was obtained by dividing the apparent density by the density of bulk SiO₂, 2.21 g/cm³.¹⁰⁾

The ϵ' , ϵ'' , μ' and μ'' values of powder samples approach those of the bulk as the relative density increases. There are no frequency dependencies of ϵ' , ϵ'' , μ' and μ'' for powder and bulk samples except for the values at lower frequencies. The values at lower frequencies are considered to contain large errors as demonstrated by the large standard deviations in the permittivity and permeability of the bulk sample at lower frequencies.

The μ' values are nearly unity irrespective of the relative densities, which is the same as that of air. This corresponds to the fact that SiO₂ does not have magnetization. The values of ϵ'' and μ'' for SiO₂ are nearly zero over the experimental frequencies. This indicates that SiO₂ has poor mi-

crowave absorptivity. The ϵ' and ϵ'' values obtained in this study are in good agreement with the reported values as detailed in the Discussion section, which confirms the feasibility of the present measurement.

3.2. Permittivity and Permeability of Fe₃O₄

Figure 3 shows the value of ϵ' and ϵ'' and Fig. 4 shows μ' and μ'' for powder and bulk Fe₃O₄ samples as a function of frequency f , respectively. The powder Fe₃O₄ sample with each relative density was measured once. The sintered and

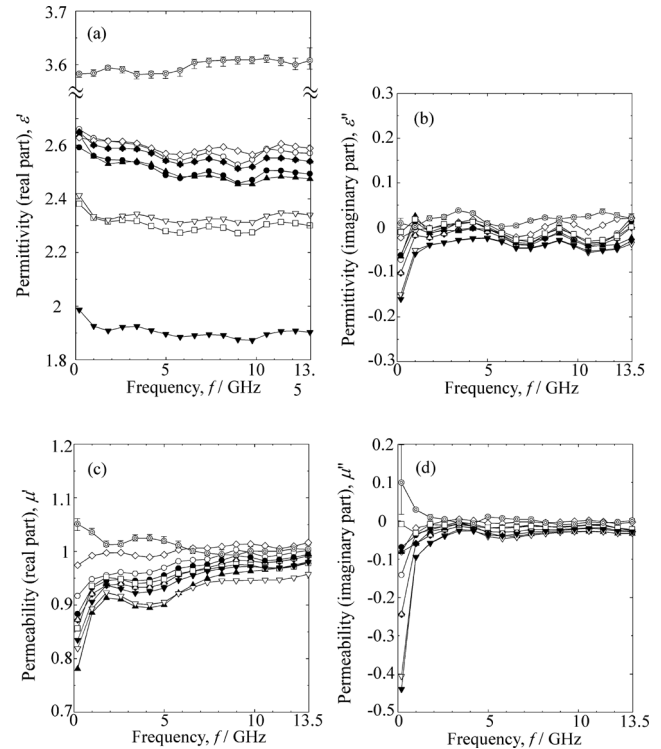


Fig. 2. Frequency dependency of complex permittivity and permeability on powder and bulk SiO₂. Relative densities of powder samples: ▼ 46.8%, ▽ 59.2%, □ 59.6%, ▲ 66.2%, ● 67.4%, △ 68.4%, ◆ 69.5%, ○ 69.9%, and ◇ 70.2%. ⊙ denotes bulk SiO₂.

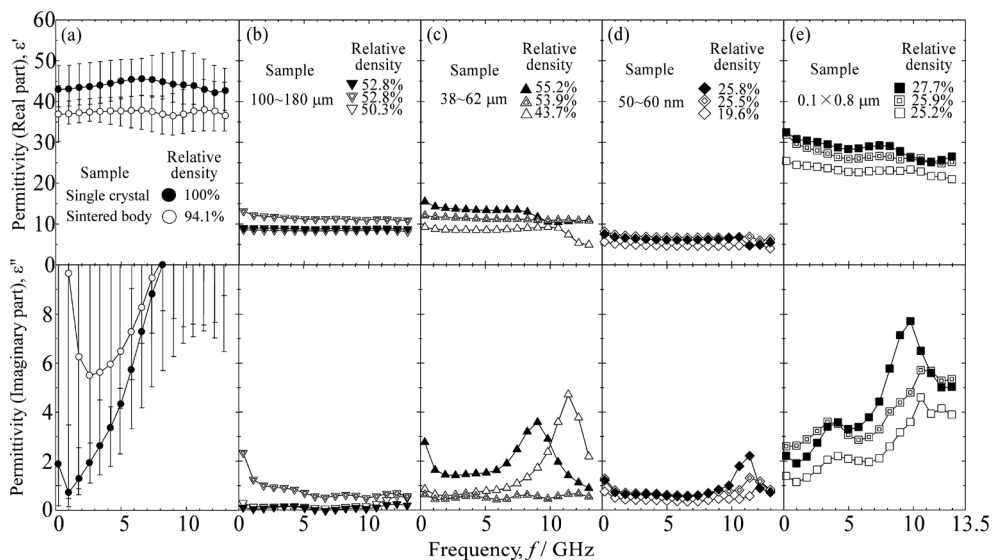


Fig. 3. Complex permittivity ϵ' and ϵ'' of powder and bulk Fe₃O₄ samples as a function of frequency. (a) a single crystal and sintered body, (b), (c), and (d) spherical powders having particle sizes of (b) 100–180 μm , (c) 38–62 μm and (d) 50–60 nm, and (e) acicular powders with the particle size of 0.1 \times 0.8 μm .

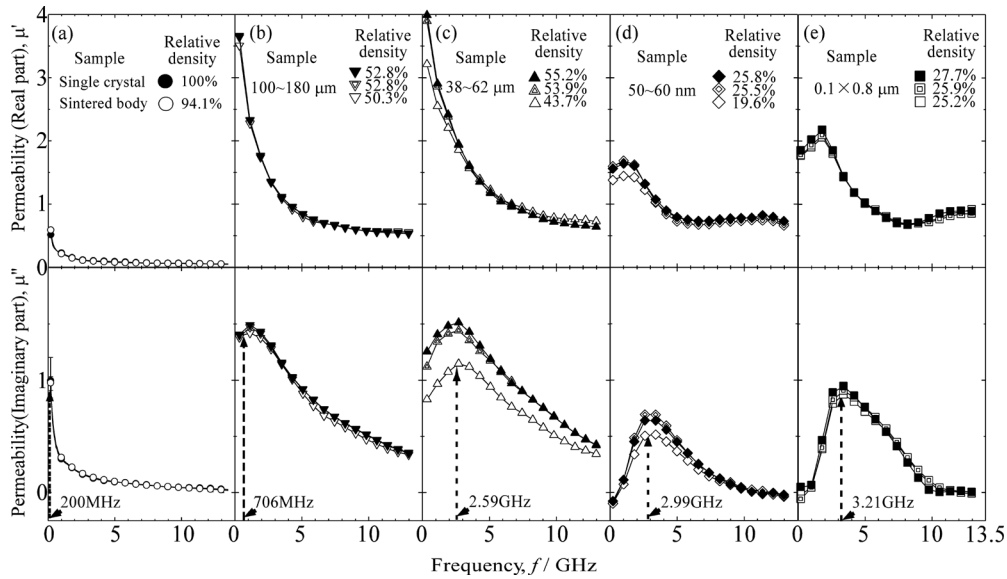


Fig. 4. Complex permeability μ' and μ'' of powder and bulk Fe_3O_4 samples as a function of frequency. (a) a single crystal and sintered body, (b), (c), and (d) spherical powders having particle sizes of (b) 100–180 μm , (c) 38–62 μm and (d) 50–60 nm, and (e) acicular powders with the particle size of 0.1 \times 0.8 μm .

single crystal Fe_3O_4 samples were measured 11 and 20 times, respectively, and the average values are plotted in Figs. 3 and 4. The relative density of a powder sample was obtained by dividing the apparent density by the density of bulk Fe_3O_4 , 5.03 g/cm³ which was determined by measuring mass and volume of the single crystal.

It can be seen from Fig. 3 that the ϵ' values of all samples are almost constant irrespective of frequency below *ca.* 8 GHz while the ϵ' values slightly decrease with an increase in the frequency above *ca.* 8 GHz. In fact, the ϵ'' values show a peak around 10 GHz just below which the ϵ' values show a slight decrement. These results indicate that Fe_3O_4 powders can be heated by the dielectric loss to some extent.

The μ' values of the bulk Fe_3O_4 and the Fe_3O_4 powders with the particle sizes of 100–180 μm and 38–62 μm monotonically decrease with increasing the frequency in the experimental frequency range, on the other hand those of the Fe_3O_4 powders with the particle sizes of 50–60 nm and 0.1 \times 0.8 μm show a peak around 1 and 2 GHz, respectively. As for the μ'' values, the values of the bulk Fe_3O_4 monotonically decrease with an increase in the frequency in the experimental frequency range while those of the Fe_3O_4 powders with the particle sizes of 100–180 μm , 38–62 μm , 50–60 nm and 0.1 \times 0.8 μm show a peak at 706 MHz, 2.59 GHz, 2.99 GHz and 3.21 GHz, respectively, as shown in Fig. 4.

4. Discussions

4.1. Relationship between Permittivity (Real Part) and Relative Density

Figure 5 shows the relation of the value of ϵ' at 5.8 GHz to the relative density for SiO_2 . The values reported by Inoue *et al.*¹¹⁾ are also included in this figure. It can be seen that the values in this study are in good agreement with the reported values. The relation between the ϵ' values and the relative densities d_r can be expressed by the equation

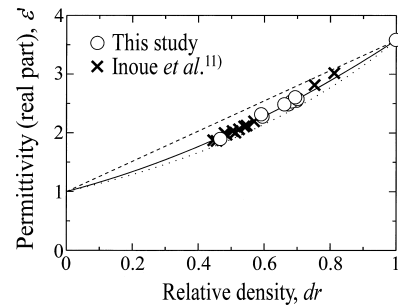


Fig. 5. Permittivity (real part) ϵ' of powder and bulk SiO_2 at 5.8 GHz as a function of the relative density d_r .

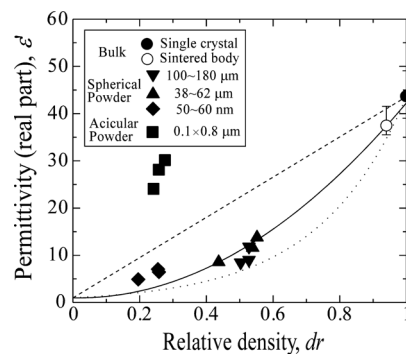


Fig. 6. Permittivity (real part) ϵ' of powder and bulk Fe_3O_4 at 2.45 GHz as a function of the relative density d_r .

$\epsilon' = 1.11d_r^2 + 1.47d_r + 1$ (solid line in Fig. 5). This indicates that the ϵ' values of powder samples with the relative density below 1 are smaller than the values estimated using the linear relation between ϵ' and d_r (dashed line), and larger than those estimated using the Lichtenecker's logarithm mixed law (dotted line)¹²⁾ described by the equation $\log(\epsilon') = (1 - d_r) \log(\epsilon'_{\text{air}}) + d_r \log(\epsilon'_{\text{SiO}_2})$ where ϵ'_{air} is the ϵ' value of air, *i.e.*, unity and ϵ'_{SiO_2} is the ϵ' value of bulk SiO_2 .

Figure 6 shows the relation of the value of ϵ' at 2.45 GHz to the relative density for Fe_3O_4 . 2.45 GHz is one

of the ISM bands, which is the most industrially-used frequency. The relation between the ϵ' values and the relative densities d_r can be expressed by the equation $\epsilon' = 42.67d_r^2 - 1.37d_r + 1$ (solid line) although the data of acicular powders are not considered for the least-square fitting. This also indicates that the ϵ' values of powder samples with the relative density below 1 are lower than the values estimated using the linear relation (dashed line) and larger than those estimated using the Lichtenecker's logarithm mixed law (dotted line). The ϵ' values of acicular powders are deviated on the upper side from the above equation and are about 4 times larger than those of spherical powders. Although the deviation does not stem from the experimental error, the reason of the deviation has not been clarified yet.

4.2. Frequency at the Peak of μ'' vs. Frequency Curve of Fe_3O_4

It can be seen from Fig. 4 that the frequencies at the peaks of the μ'' vs. frequency f curves are 1–1.5 GHz higher than the frequencies at the peak of the μ' vs. f curves for the Fe_3O_4 powders with the particle sizes of 50–60 nm and $0.1 \times 0.8 \mu\text{m}$. This result is reasonable and suggests that the peaks may also exist at the frequencies smaller than 200 MHz, the minimum of the experimental frequency range, for the Fe_3O_4 powders with the particle sizes of 100–180 μm and 38–62 μm . In this section, we have focused on the μ'' values, which is the more important parameter for microwave heating.

Figure 7 shows the relation of the frequency at the peak of μ'' - f curve to the particle size of Fe_3O_4 sample. In this figure, the peak frequency of bulk Fe_3O_4 is assumed to be below 200 MHz. This figure demonstrates that the frequency at the peak of the μ'' - f curve decreases with an in-

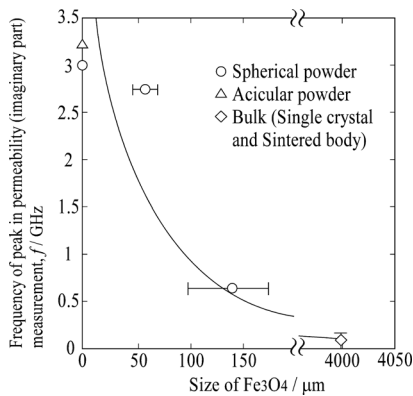


Fig. 7. Relation between frequency at the peak of μ'' - f curve and size of Fe_3O_4 sample.

crease in the particle size of Fe_3O_4 sample. In general there are two magnetization processes; one is displacement of domain walls and the other is rotation of the magnetic moments in a domain. Rado *et al.*¹³⁾ have measured the μ'' values of sintered Mg-Fe ferrite as a function of frequency and have found that two peaks exist in the radio frequency range (*ca.* 50 MHz) and in the microwave frequency range (*ca.* 2.5 GHz). They have also measured the μ'' values of fine Mg-Fe ferrite powders embedded in the paraffin wax and have found that only one peak is found at higher frequency and the peak at lower frequency does not appear. It has been experimentally found that the fine Mg-Fe ferrite powder is composed of single domain and the lack of lower frequency peak is relevant to the lack of domain wall movements. They have concluded that lower frequency peak is primarily due to the domain wall displacement and higher frequency peak is due to the rotation of magnetic moments in the domains, which is called “rotational resonance”. As for the high frequency peak, Viau *et al.*¹⁴⁾ have measured the particle size dependency of microwave permeability for $\text{Fe}_{14}\text{Co}_{43}\text{Ni}_{43}$ and have found that the μ'' vs. f curve of sub-micron ferromagnetic particles exhibit several peaks in the microwave frequency range although *ca.* 1 μm particles show only one peak in the same frequency range. They have also found that the peaks due to submicron particles are shifted to higher frequencies with decreasing particle size from 125 to 90 nm and then to 60 nm. Actually, the particle size dependency of the frequency at the peak of the μ'' - f curve for Fe_3O_4 micron particles in our study closely resembles the particle size dependency of the $\text{Fe}_{14}\text{Co}_{43}\text{Ni}_{43}$ submicron particles. However, we don't know if the mechanism of the size dependencies is really the same between the micron and submicron particles. At least, we can conclude that the peak of μ'' vs. f curve for the Fe_3O_4 powders in this study is associated with the “rotational resonance” because the peak is observed in the microwave frequency range, however, we don't know the reason of the particle size dependency so far.

4.3. Optimal Microwave Frequency for Heating Magnetite Iron Ores

Figure 8 shows the values of μ'' for powder and bulk Fe_3O_4 samples at (a) 1 GHz, (b) 2.45 GHz and (c) 12 GHz as a function of the relative densities. It is found from Fig. 8(a) that the μ'' values of the powder Fe_3O_4 samples with the particle size of 50–60 nm and $0.1 \times 0.8 \mu\text{m}$ are much lower than those of the powder Fe_3O_4 samples having larger particle size. The same results can also be obtained in the frequencies lower than *ca.* 2 GHz. On the other hand, as

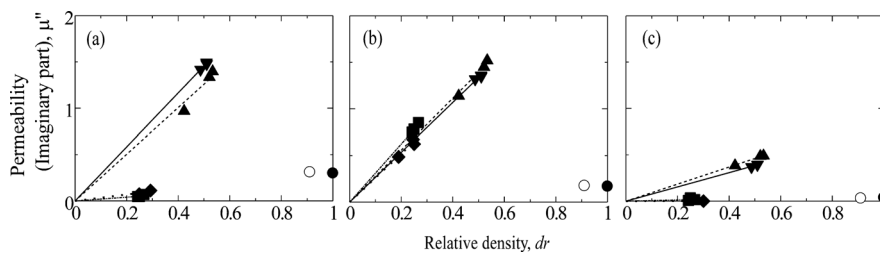


Fig. 8. Permeability (imaginary part) μ'' of powder and bulk Fe_3O_4 at (a) 1 GHz, (b) 2.45 GHz and (c) 12 GHz as a function of the relative densities. Particle sizes of powder samples: — \blacktriangledown — 100–180 μm , --- \blacktriangle --- 38–62 μm , ... \blacklozenge ... 50–60 nm, --- \blacksquare --- $0.1 \times 0.8 \mu\text{m}$. \circ and \bullet denote sintered body and single crystal, respectively.

shown in Fig. 8(b), all the powder samples have almost the same μ'' values at the constant relative densities around 2 to 3 GHz. Above *ca.* 3 GHz, the μ'' values of all samples become smaller as the measurement frequencies become higher (see Fig. 8(c)). These results correspond to the fact that the peak frequency, *i.e.*, the rotational resonance of the powder Fe_3O_4 samples with the particle size of 38–62 μm and 50–60 nm are around 2–3 GHz, as shown in Figs. 4(c) and 4(d). Now, let us consider the microwave ironmaking, the final target of our studies. Since iron ores have a wide range of particle sizes, inspection of Fig. 8 suggests that 2.45 GHz, the frequency of the most commercially prevailing microwave generators, may be the most suitable one for microwave heating of iron ores. Even if the heating due to the dielectric loss is taken into account, 2.45 GHz may be the most optimal microwave frequency as the ε'' values at 2.45 GHz is rather high.

Finally the authors would like to insist that whenever we try to develop the high temperature process using microwave, it is very important to elucidate the complex permittivity and permeability of all the materials related to the process such as raw materials, products, byproducts, refractory materials and so on.

5. Conclusions

The complex permittivity and permeability of powder and bulk SiO_2 and those of powder Fe_3O_4 with the particle size of 50 nm–180 μm and a sintered and a single crystal Fe_3O_4 were measured. The effects of various factors such as microwave frequency, relative density and particle size of powder samples on the permittivity and permeability were investigated. The obtained results are summarized as follows.

(1) The ε' values of powder SiO_2 and Fe_3O_4 samples with the relative density below 1 are smaller than the values estimated using the linear relation between ε' and the relative density, and larger than those estimated using the Lichtenecker's logarithm mixed law.

(2) The values of μ'' for all of the powder Fe_3O_4 samples show the peaks in the frequency range of 706 MHz to 3.21 GHz. The frequency at the peak of the μ'' vs. frequency curve decreases with an increase in the particle size

of Fe_3O_4 sample.

(3) All the powder samples with an identical relative density have almost the same μ'' values around 2 to 3 GHz irrespective of the particle size. Above *ca.* 3 GHz, the μ'' values of all samples become smaller as the measurement frequencies become higher. These results suggest that 2.45 GHz, the frequency of the most commercially prevailing microwave generators, may be the most suitable one for microwave heating of iron ores.

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