

*Review*

Microwave Processing and Applications to Future Automobile

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■**ABSTRACT**■ Microwave processing is expected to become an energy-saving technology and environmentally green process. This method has many advantages over conventional methods such as rapid heating, selective heating, and internal heating. Extremely rapid heating of 100 °C/s has been achieved using a newly developed cavity resonator and absorbents. A single-mode cavity that can concentrate microwave energy was developed and the possibility of rapid sintering was examined for several functional ceramics. Hydrogen production by reforming with microwave heating was also conducted as an application for the auto industry in the near future. Almost 100% conversion was successfully achieved with quick start-up and at a low temperature of 500°C. Under microwave irradiation, the activation energy for reforming could be reduced to 2/3 against that of conventional methods. The saccharification of cellulosic biomass with solid acid catalysts was also attempted under microwave irradiation. By using catalysts of carbon-based powders, high yields were obtained within short times. This method has the potential to achieve saccharification without expensive enzymes or pretreatment, which are typically required. Furthermore, the microwave heating of metal powders was investigated in detail and the heating mechanism was clarified. Non-magnetic powders such as Al and Cu were rapidly heated with fine grains and surface oxidation in a magnetic field, so it seemed due to surface resistance heating by eddy current loss. In addition, the complex permittivity of materials in the microwave region and the electromagnetic field distribution in the cavity by finite-difference time-domain (FDTD) analysis are presented.

■**KEYWORDS**■ Microwave Processing, Ultrahigh-speed Heating, Functional Ceramic Sintering, Hydrogen Production, Cellulosic Biomass, Saccharification, Non-magnetic Metal Powder, Complex Permittivity, Electromagnetic Field Distribution

1. Introduction

Microwave heating is characterized by self-heating, whereby heat is generated in a sample by the absorption of microwave power. The heating rate is extremely high compared to conventional external heating methods such as thermal conduction or radiation. The microwave process may give a large impact as future new technologies to be possible to decrease the energy consumption by approximately one-digit compared with traditional processes. The key points concerning microwave heating are the design of the cavity resonator and the selection of materials for absorption of microwaves. If a single-mode cavity of energy concentration type (heat chamber having high electromagnetic field intensity) is used, then the microwave energy not only creates advanced materials having new characteristics, but also has the possibility of use as a new automotive power source. By using these superior microwave technologies, it is necessary

to develop applications to environmental fields. In our research, the microwave heating of porous ceramics was attempted using a newly developed cavity resonator and microwave absorbents,⁽¹⁾ and the sintering of functional ceramics was examined.⁽²⁾ Microwave sintering of ceramics is expected to result in excellent electrical and mechanical properties due to the promotion of crystallinity, high densification and fine grain size.

The shift toward a hydrogen society has been increasingly accelerated as alternative energy sources for next generation devices. To realize this, it is important to propose methods for the clean production and supply of hydrogen at low cost. However, presently most hydrogen is produced from fossil fuel hydrocarbons.⁽³⁾ Recent studies have shown that hydrogen can be produced by reforming ethanol fuels made from biomass such as sugarcane, corn and crop waste.^(4,5) Biomass is expected as both a renewable energy source and means of CO₂ reduction. It is said

that the decrease in activation energy is significantly affected for the promotion of microwave reaction.⁽⁶⁾ A new method of microwave reforming was devised using a combination of bio-ethanol and microwave heating. In microwave reforming, only a catalyst layer is selectively heated from the inside over a short time, so it becomes possible to reform quickly with a simple setup and with very high energy efficiency.

Research on the production of biofuels from cellulosic resources, which do not compete with food resources, has been actively conducted. However, the saccharification of cellulose is very difficult, costly and time consuming. We have developed a new method for the saccharification of cellulosic biomass without pretreatment. It is previously known that lignin is selectively decomposed into low molecular weight when wood is irradiated with microwaves.⁽⁷⁾ Materials with hydroxyl groups, such as water or alcohol, are good microwave absorbents. Recently, research on new saccharification methods that use solid acid catalysts has advanced in various fields.^(8,9) When cellulose and solid acid catalysts containing hydroxyl groups come in contact under microwave irradiation, the glycosylation reaction is promoted, and so there is the possibility that cellulose could be efficiently decomposed and glycosylated. This method does not require pretreatment, and the cheap solid catalyst can be repeatedly used. It is expected that this method can produce biofuels or chemicals at lower cost.

The microwave processing of metal materials is expected to have applications such as surface treatment or sintering of automotive parts. Metal materials, if they are powders, can be heated by microwave irradiation.⁽¹⁰⁾ However, the types of metals heated by microwaves, the heating uniformity and reproducibility, and the heating mechanism have yet to be clarified. In this study, we investigated the effects of surface oxidation on the microwave heating of non-magnetic metal and metal oxide powders, and the heating mechanism is discussed.

Furthermore, as microwave basic technologies, dielectric properties of materials were investigated and calculations of the electromagnetic field distribution in the cavity were performed. It is very important to understand the complex permittivity and permeability of materials for microwave heating. In the literature, a detailed investigation of the complex permittivity of materials has been reported by Hippel⁽¹¹⁾ and others; however, since then there has been no systematic

data published. Therefore, it is necessary to prepare a database of permittivity and permeability for various materials. The important factors in microwave heating are the imaginary parts, i.e., the dielectric and magnetic loss factors. It is important to understand the frequency and temperature dependence of these powders and liquids for the selection of a suitable frequency and cavity design, and to achieve uniform heating. It is necessary to understand the electromagnetic field distribution in the cavity to uniformly heat a sample under microwave irradiation. The electromagnetic field distributions of a multi-mode cavity are much more complex than a single-mode cavity. The electromagnetic field distribution in a multi-mode microwave oven was analyzed by the finite-difference time-domain (FDTD) method, and the electric field intensity distribution was actually measured using a photoelectron field sensor.

2. Principle and Characteristics

When dielectric and magnetic materials are placed in an electromagnetic field, the dipoles, ions and hysteresis loss in these materials cause electron or magnetic spin by the vibration of microwave radiation.⁽¹²⁾ The energy absorbed in heating per unit volume P , is given by the Maxwell equation:⁽¹³⁾

$$P = 1/2 \cdot \epsilon_0 \cdot \epsilon'' \cdot \omega \cdot E^2 + 1/2 \cdot \sigma \cdot E^2 + 1/2 \omega \cdot \mu_0 \cdot \mu'' \cdot H^2, \quad (1)$$

where ϵ_0 is the dielectric constant of a vacuum, ϵ'' is the dielectric loss factor (the imaginary part of the permittivity), ω is the angular frequency, σ is the electric conductivity, E is the electric field intensity, μ_0 is the magnetic constant of a vacuum, μ'' is the magnetic loss factor (the imaginary part of the permeability), and H is the magnetic field intensity. The first term is the dielectric loss by permittivity (that is dielectric heating), the second term is the conduction loss by conductivity (electric resistance heating), and the third term is the magnetic loss by permeability or susceptibility (magnetic heating).

The dielectric loss factor ϵ'' changes with temperature and frequency. The dielectric properties at high temperature are very important in heating ceramics with microwave irradiation. The value of ϵ'' is generally small in ceramics at room temperature, but it rapidly increases with temperature. For example, the ϵ'' increases exponentially with increasing temperature

in alumina, and ϵ'' at 1800°C becomes over 100 times higher than that at room temperature, as shown in **Fig. 1**.⁽¹⁴⁾ Therefore, once microwave heating starts, the sample is rapidly heated without high power, because the absorption increases with temperature. For that reason, microwave heating is expected to promote the sintering and joining of ceramics.^(15,16) **Figure 2** shows the differences between microwave

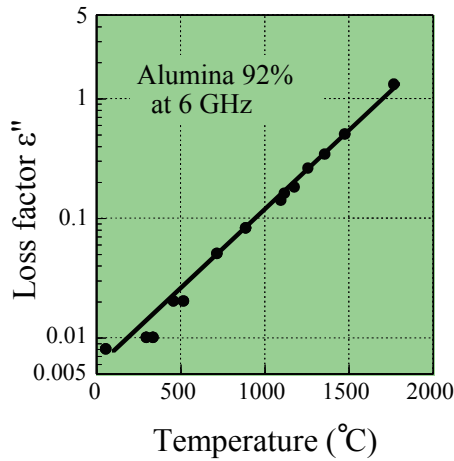


Fig. 1 Temperature dependence of dielectric loss factor for alumina ceramics.

and conventional heating. It is anticipated that a liquid phase at the grain boundaries contributes to microwave heating. At the microscopic level, this phase, which has a large loss factor, is preferentially heated and enters a thermal non-equilibrium state. It is presumed that it will be apparently the low temperature state on the macro-level actually measuring the sample temperature. As a result, heating at low temperature for a short time can be achieved with microwave heating. In contrast, conventional external heating methods result in uniform heating at the microscopic level, but non-uniform heating at the macroscopic level, as opposed to microwave heating.

Microwave processing has many advantages over conventional heating methods, and can lead to the creation of advanced materials and to new material processing technologies. However, before this processing can be practically applied, there are some problems that must be solved. These include uneven heating by thermal runaway,⁽¹⁷⁾ the high sensitivity of heating to the powder composition, and the limited sample size. The key points of this processing are dependent on how these problems are solved, while making use of the best characteristics of microwave heating.

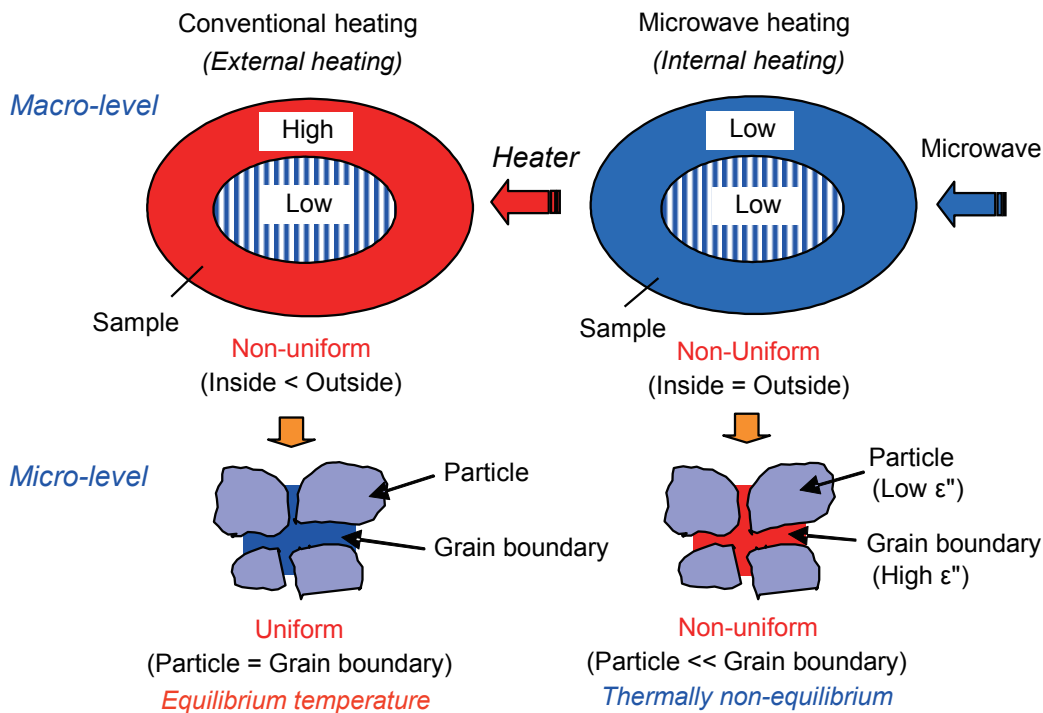


Fig. 2 Differences between microwave and conventional heating.

3. Applications to Future Automobile

Applications of microwave processing in the auto industry have been limited to rubber vulcanization and ceramic drying until now. New technologies that take full advantage of the characteristics of microwave heating are expected. **Figure 3** shows applications of microwave heating expected in the near future; mass production of various materials such as ceramics, polymer and metal powders, heating power supplies for application such as fuel cells or electric vehicles, and environmental treatment or purification of emission gases or metal recovery. To achieve these technologies, it is necessary to develop new microwave processing with power saving and rapid heating systems that replace traditional electric furnaces. As future technologies that employ microwave processing, hydrogen production by fuel reforming, next-generation bio-fuels from cellulosic biomass, carbon fiber reinforced plastic (CFRP) curing, and catalyst synthesis are expected. There are three main characteristics of microwave processing: rapid heating, selective heating and internal heating. **Figure 4** shows profiles for the sintering of ceramics by microwave and conventional processing. In microwave processing, heating speeds that are 50 times or more higher than those with conventional methods are obtained, and energy consumption is an order of magnitude lower than that with external heating using traditional electric furnaces. This method can thus be expected to improve productivity, and reduce both the heat treatment time and environmental load using small-scale on-site microwave processing facilities.

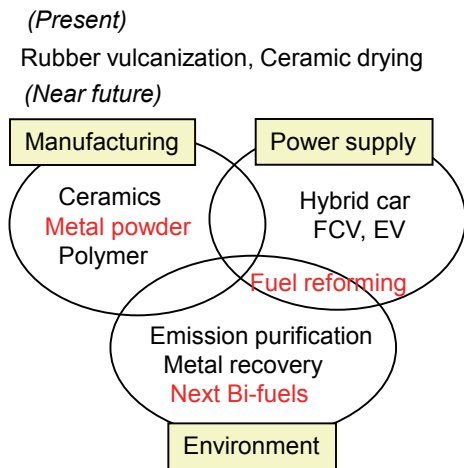


Fig. 3 Future applications of microwave heating for the auto industry.

3.1 Ultrahigh-speed Heating

A remote-control system was developed to precisely control the temperature and microwave power.⁽¹⁸⁾ This system has a single-mode cavity that can concentrate the microwave energy. The rectangular single-mode TE₁₀₃ cavity is shown in **Fig. 5**. Heating was conducted at the position of maximum electric field intensity. The cavity was turned to a resonant state by moving a coupling window (iris) for introduction of the microwave power and a short circuit plate (plunger). The iris and plunger were controlled during sample heating so that the cavity was always in a resonant state. Magnetron oscillator of 2.45 GHz was used in this system. Various conventional SiC powders and new materials were used as microwave absorbents, and they were coated into porous ceramics.

The heating rate with the single-mode cavity was compared to that with a multi-mode cavity used as a microwave oven. **Figure 6** shows the heating profiles for each cavity and an in-situ observation of a porous ceramic coated with new absorbents. A ceramic monolith 30 mm in diameter coated with

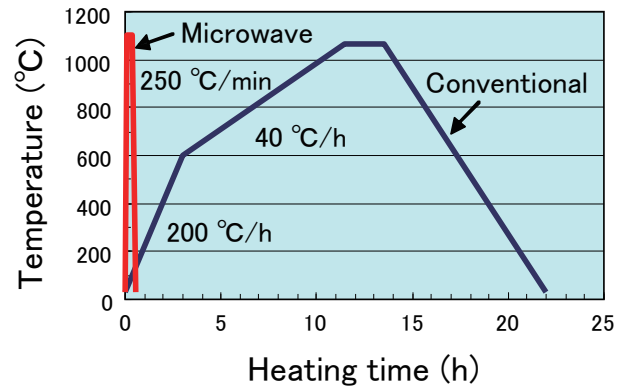


Fig. 4 Heating profiles for microwave and conventional sintering of ceramics.

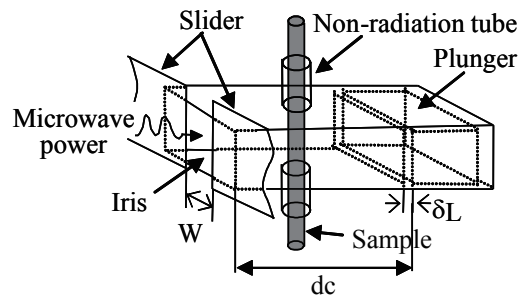


Fig. 5 Single-mode rectangular cavity.

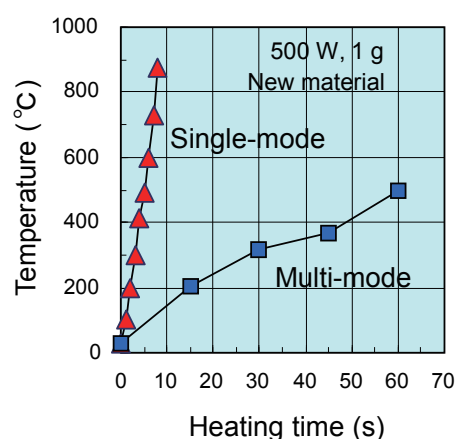
new absorbents was heated up to 1000°C from room temperature in 10 s. Thus, an extremely high heating rate of 100 °C/s was achieved using the developed cavity and absorbents. In the sample coated with conventional SiC powder, thermal runaway, i.e., the phenomenon where the temperature runs out of control, occurred from approximately 500°C, and the sample was then unevenly heated. In contrast, the sample coated with the new absorbents was repeatedly and stably heated to high temperatures. The new absorbents exhibited extremely large dielectric loss factors (i.e., high microwave absorption) compared to conventional SiC. The heating rate with the single-mode cavity was more than 10 times that with the multi-mode cavity. This method is expected to be used as a cold emission measure for exhaust gas purification.

3.2 Microwave Sintering of Functional Ceramics

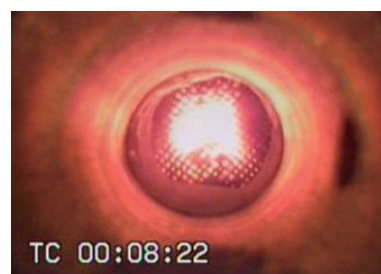
As functional ceramics, ZnO varistors, lead zirconate titanate (PZT) actuators and capacitor built-in multilayer were sintered with the microwave process.⁽¹⁹⁾ Microwave sintering of ZnO was relatively easy and the theoretical density was reached. Several types of additives were added to ZnO to control the varistor properties. The powder was molded into a cylindrical compact (9 mm diameter, 7 mm long) and rapidly heated at a rate of ca. 300 °C/min at 6 GHz using the single-mode cavity. The density, grain size, varistor voltage and non-linearity factor α , were measured.

A higher α was achieved than that with conventional sintering. The high varistor voltage was ascribed to the small grain size obtained in the microwave-sintered material. We consider that the preferential heating of the additives contributes to the excellent properties.

Local heating occurred in PZT due to thermal runaway. A new method was developed to prevent this uneven heating, in which the PZT sample was placed between SiC sheets with high microwave absorption. A large-scale cavity (2.45 GHz) was used for the sintering of PZT.⁽²⁰⁾ The successful prevention of thermal runaway is attributed to the higher absorption and lower temperature dependence of the loss factor in SiC than in PZT. The silicon carbide sheets relaxed the rapid change of the loss factor, and the PZT sample was preferentially heated to a high temperature. Microwave sintering was performed at a heating rate of 170 °C/min in air. The PZT pellet with a diameter of 16 mm was uniformly sintered. The same sample was also sintered in an electrical furnace at a heating rate of 300 °C/h for 4 h in a PbO atmosphere. With microwave sintering, a high density exceeding 8.2 g/cm³ was attained by sintering at low temperature for a short time (900°C, 5 min). This density was higher than that obtained by conventional sintering. The maximum evaporation was only 0.3% for microwave sintering, even in a PbO-free atmosphere. **Figure 7** shows X-ray diffraction patterns and the grain size-dependence of PZT obtained by microwave and conventional sintering. For microwave sintering at 900°C for 5 min, the pyrochlore phase in the raw materials disappeared

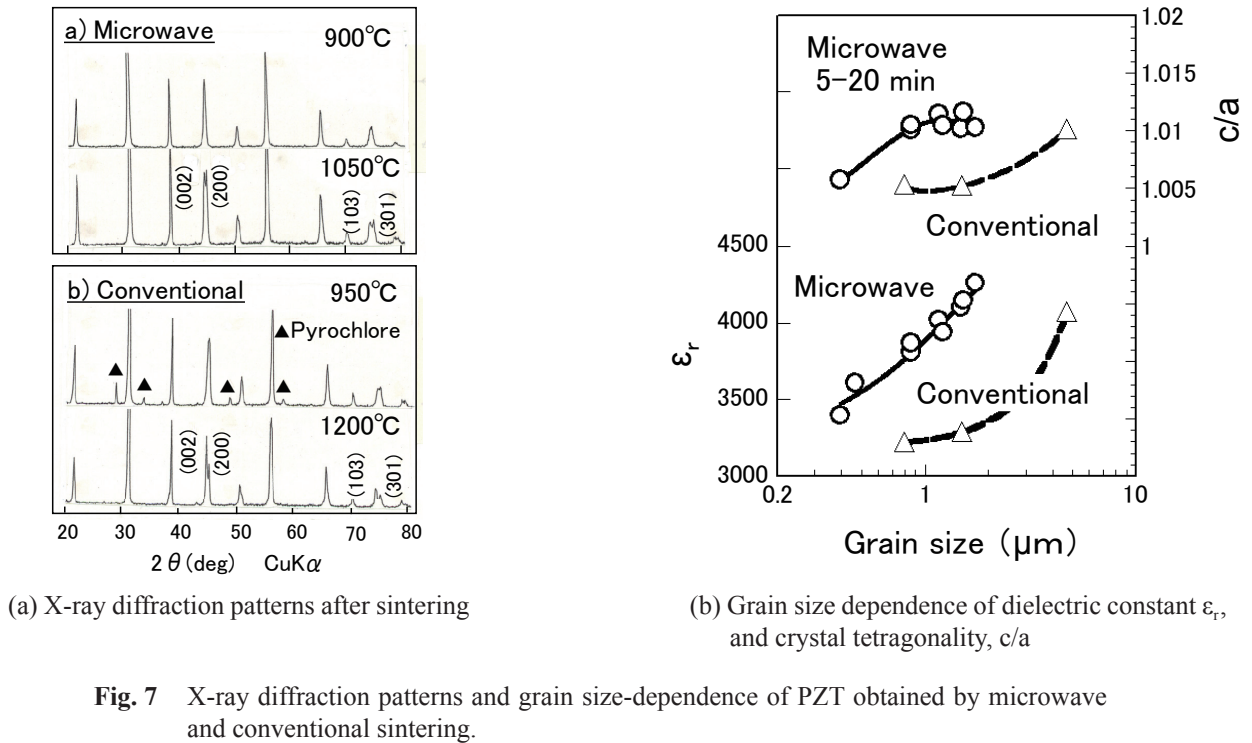


(a) Comparison of single-mode and multi-mode cavities at 2.45 GHz.



(b) In-situ observation of single-mode cavity with a CCD camera

Fig. 6 Heating profiles for porous ceramics coated with new absorbents.



and only the perovskite phase was present, as shown in Fig. 7(a). When the sintering temperature was raised to 1050°C, split peaks associated with a tetragonal structure appeared. In conventional sintering, the pyrochlore phase was noted at 950°C for 4 h, which indicates that sintering was not completed. The peak splits were not observed below 1200°C with conventional sintering. It was considered that the sinterability was clearly promoted with microwave sintering. The grain size-dependence of the dielectric constant ϵ_r , and the crystal tetragonality c/a , calculated from the lattice constants are shown in Fig. 7(b). For conventional sintering, ϵ_r did not increase until the grain size was about 5 μm . For microwave sintering, ϵ_r rapidly increased with the grain size, and a high ϵ_r was obtained even with a grain size of 1.5 μm . Similarly, c/a was large for a grain size of 1 μm with microwave sintering, which clearly indicates the promotion of crystallinity. The strength was improved by ca. 15% for the same displacement with microwave sintering over that obtained with conventional sintering. When comparing electric properties such as the piezoelectric constant d_{31} , and the dielectric constant ϵ_r , the strength increase of ca. 30% was observed compared to those for conventional sintering. Thus, excellent piezoelectric and dielectric characteristics were obtained with microwave sintering.

In the simultaneous sintering of multilayer ceramics with built-in capacitors, interfacial delamination in the electrode is a serious problem. In particular, the dielectric constant ϵ_r is significantly decreased when the glass elements in the substrate diffuse into the dielectric. Microwave heating may reduce the diffusion between the substrate and dielectric by sintering at lower temperatures for shorter times. Microwave sintering was examined with a heating rate of 100 °C/min and a sintering time of 5 min.⁽²¹⁾ For comparison, specimens were also sintered conventionally under similar conditions. In the sample, the dielectric was sandwiched between an internal electrode and the substrate. A maximum ϵ_r of 6000 was obtained by microwave sintering, without influence of the electrode thickness. With conventional sintering, the substrate was significantly deformed with a thick electrode and ϵ_r was significantly decreased with a thin electrode. The waviness by microwave sintering was only 1/100 of that by conventional sintering. **Figure 8** shows electron probe microanalysis (EPMA) results for the multilayer ceramics. With conventional sintering, the glass elements, such as Si, diffused into the dielectric, and Ag in the electrode diffused into the substrate. The crystallization temperature of the substrate decreased with the diffusion of Ag and the deformation of the substrate increased. With

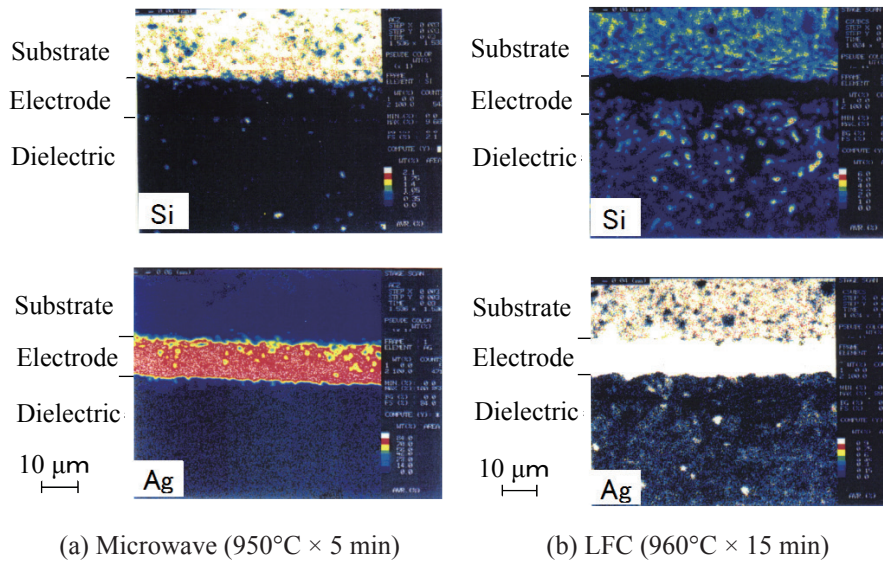


Fig. 8 Cross-sectional EPMA results for multilayer ceramics.

microwave sintering, the diffusion of glass elements into the dielectric was suppressed and there was little diffusion of Ag into the substrate. Consequently, sintering without deformation was achieved. Thus, microwave sintering can suppress diffusion, accelerate the sintering reaction, and prevent substrate deformation.

3.3 Hydrogen Production by Microwave Reforming

Microwave reforming of ethanol was attempted using a developed heating system.⁽²²⁾ The reactor vessel, which consists of a quartz tube containing catalyst pellets, was inserted in the cavity, and microwave heating was then performed (**Fig. 9**). Rh/CeO₂ supporting 5 wt% rhodium was used as the catalyst. The catalyst pellets were produced from the mixed materials, which contained Rh/CeO₂ powders and new absorbents. One gram of catalyst pellets (10 mm diameter, 8 mm thick) was inserted into the reactor. The steam reforming of ethanol can be stoichiometrically expressed by the following equation:



This reaction is an endothermic process (203 kJ/mol, 500°C), and thus requires energy from the outside. After a mixed solution of ethanol/water from a syringe pump was passed through an evaporator, it was injected into the reactor and steam reforming

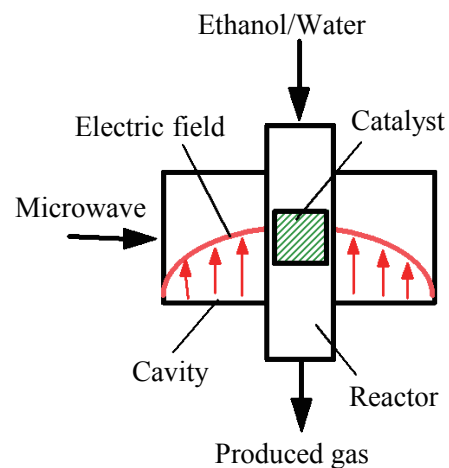


Fig. 9 Schematic diagram of microwave reforming using single-mode rectangular cavity as heat chamber.

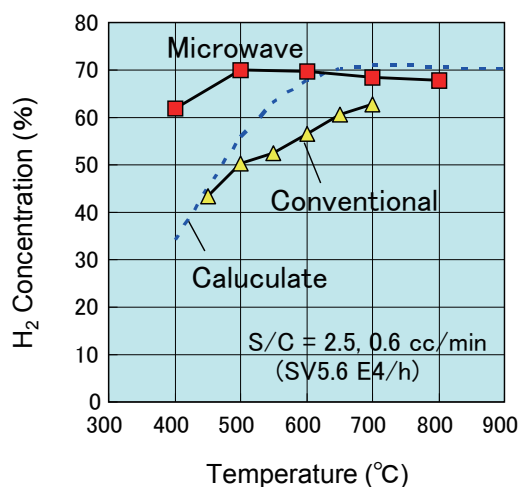
occurred. The produced gases were analyzed using gas chromatography. The influences of the steam to carbon (S/C) ratio and the gas hourly space velocities (SV) were examined. The S/C ratio of 1.5 corresponds to the ideal reaction in Eq. (2). The SV was defined as the volume ratio of evaporated gas (ethanol and water) and catalyst. Reforming by conventional external heating with an electric heater was also conducted and the advantages of the microwave method over the conventional method were examined.

The catalyst layer was quickly and stably heated under microwave irradiation. Hydrogen gas was produced within 10 s, and a constant production rate was obtained within 20 s. **Figure 10** shows

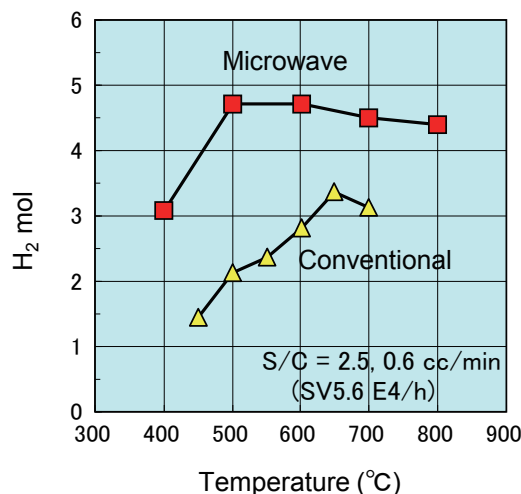
the temperature dependence of the hydrogen gas production by microwave and conventional reforming. The hydrogen concentration obtained by microwave reforming was a maximum of 70% at a low temperature of 500°C. The measured hydrogen concentrations were more significantly increased at low temperatures than those calculated based on the gas equilibrium concentration (dotted lines). On the other hand, conventional reforming required higher temperatures than the calculated values. The microwave process has the potential to significantly decrease the reforming temperature. The average temperature of the catalyst layer was low, but microscopically, a high temperature exists at the catalyst particle interfaces by selective microwave heating (see Fig. 2). Accordingly, it seems that the chemical reaction is accelerated under a thermal non-equilibrium state, so that ethanol reforming could be achieved at lower temperatures. A large amount (4.7 mole) of H_2 was obtained from 1 mole of C_2H_5OH at 500°C by microwave reforming. The hydrogen production by microwave heating was more than twice that with the conventional process at 500°C. In the conventional method, the amount of hydrogen increased with temperature, but only 3.4 moles of H_2 was formed at a high temperature of 650°C, which was the same level as that previously reported with the same type of catalyst.⁽⁵⁾

Figure 11 shows the influences of the space velocities on the hydrogen conversion and activation energy. The conversion rate is expressed as the carbon ratio of the output product gases to the input ethanol

volume. For typical ethanol reforming, full conversion is reported to be obtained above 700°C.⁽⁵⁾ However, the conversion rate with microwave reforming was already 100% at the low temperature of 500°C. In conventional reforming, it is difficult to completely resolve the ethanol, especially when the SV is increased, where the conversion tends to significantly decrease. The hydrogen production obtained with microwave reforming was three times that for the conventional method at higher SV (above 82000 /h). In addition, the activation energy with microwave reforming could be reduced to 2/3 that with the conventional method. During microwave reforming, the total gas and hydrogen gas produced increased linearly with the flow rate of the ethanol/water mixture. 1.4 L/min of total gas (920 mL/min of hydrogen gas) was obtained for a flow rate of 1 mL/min, i.e., 4.7 moles of hydrogen per mole of ethanol was obtained at high space velocities. The reformer efficiency calculated from the actual heat balance was ca. 80% on a high heating value (HHV) basis, which is approximately twice as large as that for the conventional method. High efficiency is obtained using microwaves because the catalyst layers selectively absorb the microwave energy and the particle interfaces are activated. During microwave reforming, high performance was obtained than those used for conventional reforming, especially at lower temperatures. Thus, the experimental results confirm the advantages of microwave reforming over conventional reforming.



(a) Hydrogen concentration



(b) Hydrogen production

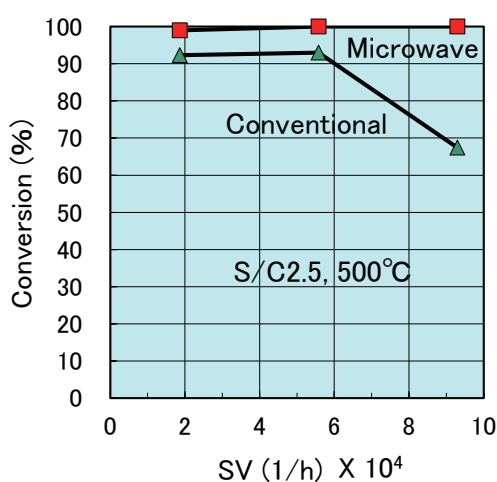
Fig. 10 Temperature dependence of hydrogen gas production by microwave and conventional reforming.

3.4 Microwave Saccharification of Cellulosic Biomass

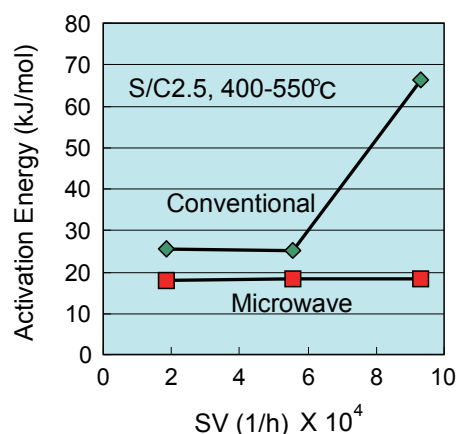
A microwave reactor device with a single-mode energy concentrating cavity was used to glycosylate raw powders of cellulose.⁽²³⁾ The frequency and maximum output power of the magnetron oscillator were 2.45 GHz and 400 W, respectively. A closed reaction vessel containing a sample was placed in the position of maximum electric field intensity. Distilled water was added and the cellulose powders were mixed with the solid acid catalysts using a magnetic stirrer. Several types of zeolite, solid superacid and carbon-based powders were used as catalysts. The saccharification of actual biomass was then attempted. Several biomass feedstocks such as bagasse, eucalyptus and cryptomeria were investigated. The average

particle size in these powders was 2-4 mm (coarse powder) and 200 μm (fine powder). For comparison with the conventional method, the influence of the heating temperature and treatment time were investigated using an oil bath. The saccharification rates for cellulose and biomass were measured with a biosensor, and the sugar components were analyzed using high-performance liquid chromatography (HPLC).

Hydrolysis saccharification of cellulose under microwave irradiation was performed with various solid acid catalysts. **Figure 12** shows the saccharification rate (yield) for cellulose under heating at 210°C for 30 min. The cellulose is converted into glucose. The results showed that it was possible to achieve the saccharification of cellulose within a short time and under low pressure compared with that



(a) Relation between conversion and SV



(b) Relation between activation energy and SV

Fig. 11 Influence of space velocities (SV) on hydrogen conversion and activation energy.

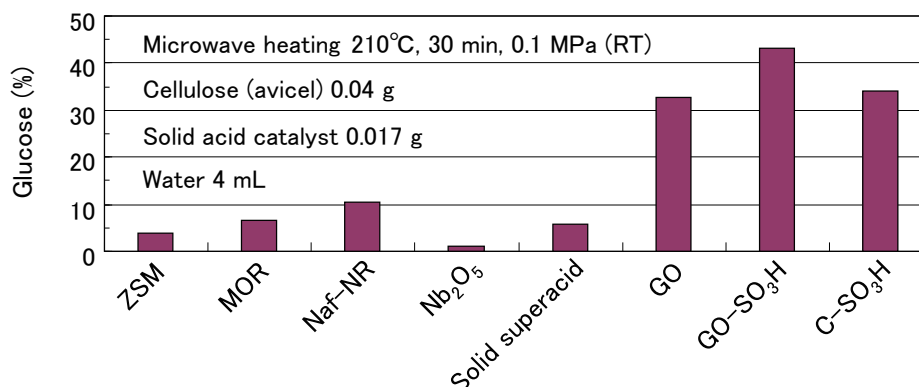


Fig. 12 Saccharification of cellulose by microwave heating with various solid acid catalysts.

previously reported for the autoclave method.⁽⁸⁾ The yield was increased to 33% using a graphite oxide (GO) catalyst that is appropriate for the microwave method, and the yield with sulfonic acid-modified GO catalyst (GO-SO₃H) was a maximum at 43%. Thus, saccharification could be achieved within an extremely short time by the microwave process. HPLC analysis indicated that most of the product was glucose with the GO catalyst, and some cellobiose was detected with the zeolite catalysts. **Figure 13** shows a comparison of the oil bath and microwave methods, which were conducted with a mesoporous silica catalyst based on organic sulfonic acids. It was difficult to achieve saccharification in the oil bath, even when heated for more than 1 hour. In contrast, high yields were obtained with the microwave process.

Microwave saccharification of actual biomass was conducted as a next step.⁽²⁴⁾ The yields of glucose and xylose were calculated as conversion rates from cellulose and hemicellulose, respectively. Almost the same yields were obtained with the GO and sulfonated carbon based (C-SO₃H) catalysts. Both catalysts gave high yields with a maximum of 50% glucose and 60% xylose using bagasse as the biomass feedstock. The same yields were achieved until the solvent concentration of 10%, as it is actually used. These catalysts were superior to other catalysts such as zeolite, solid superacid, charcoal and activated carbon. The GO catalyst has carboxylic acid groups and the sulfonated carbon has sulphonic acid groups. These catalysts have the same crystal structure

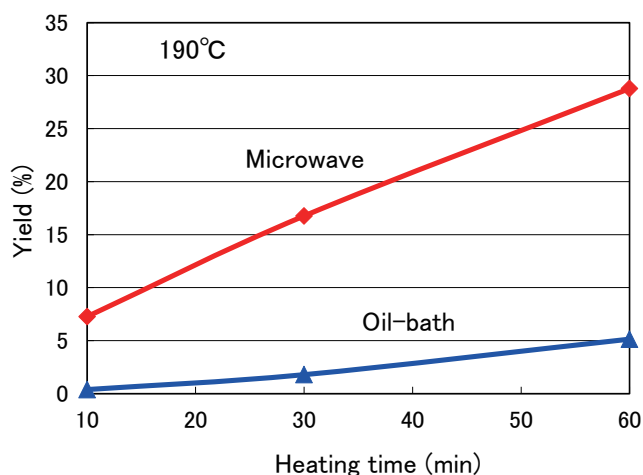


Fig. 13 Comparison of cellulose saccharification by oil bath and microwave heating with organic sulfonic acid catalysts.

as graphene. For high-yield saccharification, it is important to have Brønsted acid sites in the presence of water and hydrophilic ions. The yield of glucose increased at high temperatures, whereas the yield of xylose was a maximum at 160°C. With biomass, the microwave process has more advantages at low temperatures of 30°C and with processing times less than half of those with the oil bath method. **Figure 14** shows a comparison of microwave process using fine (200 μm) and coarse (2-4 mm) eucalyptus powders. The fine powders were obtained by ball-milling the coarse powders. The microwave saccharification was performed with sulfonic carbon under heating for 30 min. Almost the same yields were obtained for both the fine and coarse powders. In the case of enzyme treatment, saccharification is difficult with coarse powders; therefore, a pretreatment step is necessary before saccharification.⁽²⁵⁾ In contrast, the microwave process achieves saccharification without pretreatment steps such as mechanical milling. The microwave process does not use harmful chemicals, but only the recycling of inexpensive catalysts. Therefore, it is expected that microwave saccharification will replace the conventional sulphuric acid or enzymatic methods.

3.5 Microwave Heating of Non-magnetic Metal Powders

The experimental cavity can separate the electric field and magnetic field at 2.45 GHz, as shown in **Fig. 15**. A compacted powder sample (9 mm diameter)

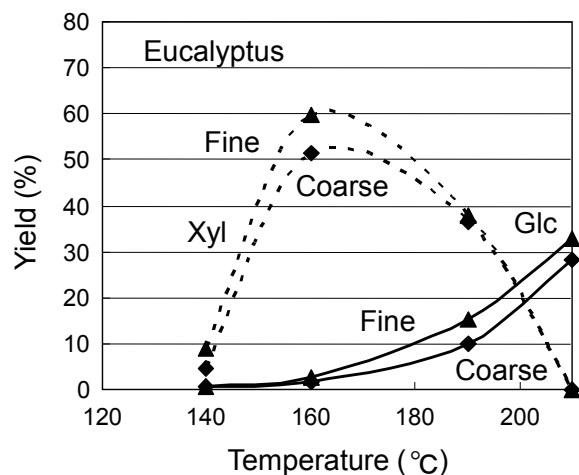


Fig. 14 Comparison of saccharification using fine and coarse eucalyptus powders. (microwave heating for 30 min, sulfonic acid carbon catalyst)

was inserted into a silica tube and heated either at the position of maximum electric (E) or magnetic (H) field intensity.⁽²⁶⁾ The surface temperature of the sample was measured with a fiber pyrometer. Microwave heating of various metallic and metal-oxide powders was performed in air or nitrogen gas, and the influence of the electric and magnetic fields on the heating characteristics was examined. Pure iron powder was easily heated in the magnetic field, although not to above the Curie point. However, it could not be heated in the electric field under nitrogen gas. Pure copper powder could not be heated in either the electric or magnetic field under nitrogen gas. However, magnetite (Fe_3O_4) and hematite (α - and γ - Fe_2O_3) iron oxide powders were rapidly heated in the electric field, but non-magnetic α - Fe_2O_3 could not be heated in the magnetic field. Metallic powders were not heated in the electric field under a reducing atmosphere, and non-magnetic powders were not heated in the magnetic field. The former result indicates that the loss by the electric conductivity was small. The latter result, where only ferri- or ferromagnetic powders were heated in the magnetic field, indicates not resistance heating due to eddy current loss, but electronic or magnetic spin heating by hysteresis loss. **Figure 16** shows microwave heating of several metallic powders in the magnetic field under the same conditions. Powders with an average particle size of $3\ \mu\text{m}$ were heated in a vacuum. Pure iron (Fe) was gradually and stably heated, but not to above the Curie point. Aluminum (Al) was rapidly heated, and titanium (Ti)

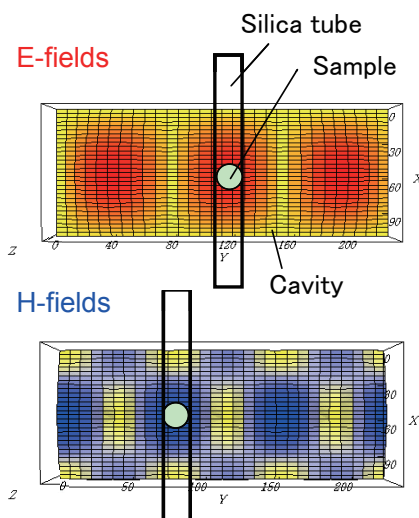


Fig. 15 Electromagnetic field distributions in single-mode cavity (TE_{103} mode).

was heated in the same manner as Fe. Copper (Cu) was only slightly heated, and was not heated over 400°C .

In further investigations, aluminum powders were heated in the electric and magnetic fields.⁽²⁷⁾ The Al powders were easily heated when the particle size was small. They were quickly and stably heated in the magnetic field, but were unstable and easily discharged in the electric field. It is considered that the effect of the microwave penetration depth (skin depth in the case of a metal) in the powder surfaces were large. In addition, they were not heated to more than the melting point and were difficult to reheat. It was supposed that the natural oxide on the powder surface was destroyed and then the microwave power did not penetrate to the inside of the sample. Therefore, surface oxidation and the connection among powders has a significant influence on the heating characteristics.

Next, the influence of the atmosphere on microwave heating was investigated for the compacted Al powders. The sample after heating in the vacuum was mostly contracted and the relative density was 93%. Liquid phases appeared at the center of the samples heated in a vacuum and under argon (Ar) gas. On the other hand, the densification of samples did not proceed without liquid phases in N_2 or air. Densification of the samples is considered to be related to the dielectric constant of the gas. The dielectric constant of gases is high for nitrogen or air, and is lowest for a vacuum. The dielectric constant is inversely proportional to the electric field intensity. Therefore, the electric field intensity in a vacuum was the highest, and thus the

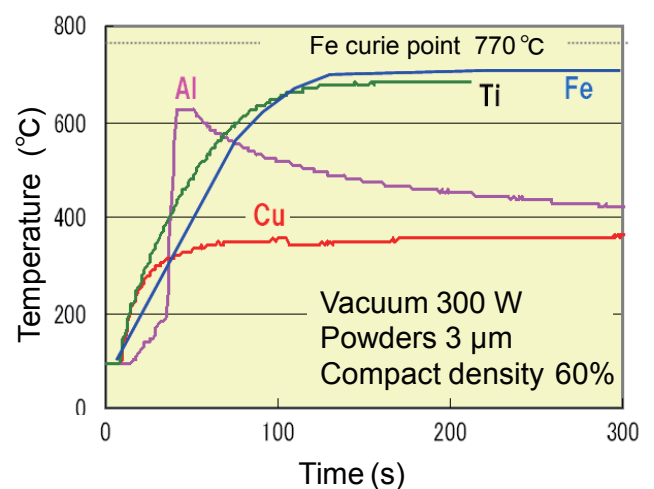


Fig. 16 Microwave heating of several metallic powders in magnetic field.

highest sample shrinkage occurred in this atmosphere.

Copper powders with different surface oxidation states were prepared. Surface-oxidized Cu foils were also used, and the surface products and oxygen distributions were analyzed. The heating profiles of surface-oxidized copper and copper oxide powders are shown in **Fig. 17**. The copper (II) oxide (CuO) powders were the most easily heated, but the copper (I) oxide (Cu₂O) powders were the most difficult to heat. The heating characteristics of the copper oxide changed significantly with the elemental valence. In Cu powders, when the amount of surface oxygen increased, they became easy to heat. Cu powders oxidized at 250°C were rapidly heated to a high temperature of 900°C, whereas Cu powders oxidized at 100°C were difficult to heat compared to the untreated Cu powders. For analysis of the surface oxidation, three different Cu foils, surface-oxidized at 100°C and 350°C, and oxide-free (acid wash) were used. The heating profiles for these foils changed significantly with the surface oxidation state, as observed for the powders. The Cu foil oxidized at 350°C was rapidly heated to over 1000°C, whereas the foil oxidized at 100°C was more difficult to heat than the oxide-free foil. **Figure 18** shows the surface products for the oxidized Cu foils measured using X-ray photoelectron spectroscopy (XPS). CuO was detected in the foil oxidized at 350°C, and Cu₂O was detected in the foil oxidized at 100°C and in the oxide-free foil. Glow discharge optical emission spectroscopy (GD-OES) analysis indicated that

the thickness for the foil oxidized at 100°C and for the oxide-free foil was very thin (less than 0.5 μm), whereas the oxide thickness for oxidation at 350°C was ca. 10 μm. Surface analyses using XPS and GD-OES indicated that when the Cu foil forms a thick CuO, the sample is rapidly heated, but when the Cu foil forms a thin Cu₂O film, then it is more difficult to heat. The Cu₂O film thickness by oxidation at 100°C was larger than that for the oxide-free foil. For this reason, the foil oxidized at 100°C was more difficult to heat than the oxide-free foil. The complex permittivity and permeability of copper and copper oxide powders were measured with the perturbation method by a resonant cavity.⁽²⁸⁾ The results indicated that the microwave heating of Cu powders is strongly influenced by ε'', and the presence of CuO is necessary.

Nonmagnetic powders such as Al and Cu were rapidly heated when fine grained and with surface oxidation, which is due to surface resistance heating by the eddy current loss in the magnetic field. The effects of the penetration depth and the surface oxidation of powders are thus very important for the heating of nonmagnetic metal powders.⁽²⁹⁾

4. Microwave Basic Technologies

4.1 Complex Permittivity in the Microwave Region

The dielectric properties of materials are very important factors for microwave heating. **Figure 19** shows the relative dielectric constant and the loss

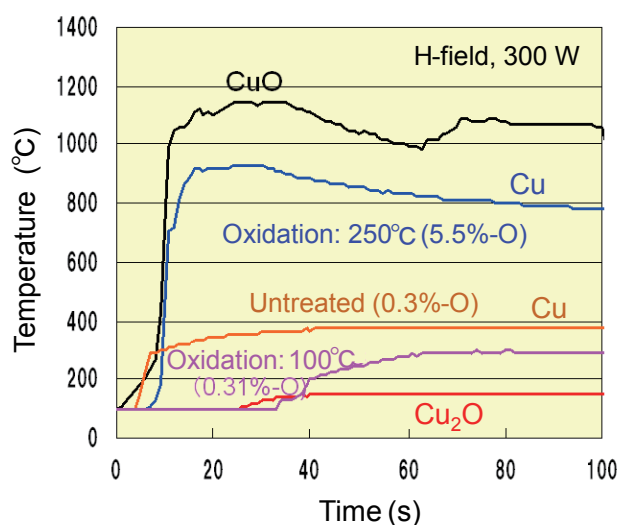


Fig. 17 Microwave heating of surface-oxidized Cu and Cu oxide powders.

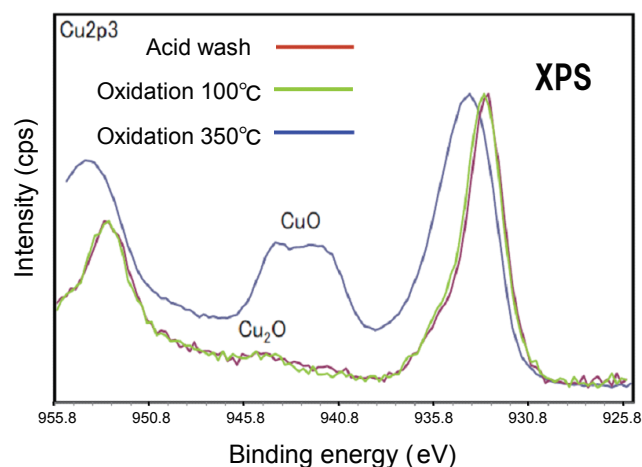


Fig. 18 XPS spectra of surface products on oxidized Cu foils.

factor for powder materials and liquids in the microwave region. The complex permittivity for various powders was measured at 6 GHz using the perturbation method (Fig. 19(a)).⁽³⁰⁾ The loss factor for pure carbon was five orders of magnitude larger than that for quartz. The loss factors for oxide ceramics were significantly different and dependent on the elemental valence. The order of values for the iron oxides was Fe_3O_4 (magnetite) > FeO (wustite) >> Fe_2O_3 (hematite). The MnO_2 and Fe_3O_4 oxide ceramics had especially large loss factors. These powders are materials that easily absorb microwave power. The ease of microwave heating is dependent on the elemental valence and the crystal structure. The heating temperature is linearly proportional to the loss factor for dielectric materials in a microwave field. In addition, the complex permittivity of typical liquids at 2.45 GHz was obtained from the literature (Fig. 19(b)).⁽³¹⁾ The loss factors for polar liquids were approximately five orders of magnitude larger than those for non-polar liquids. Polar liquids with hydroxyl groups, such as water and alcohol, have extremely large loss factors. In contrast, hydrocarbons such as gasoline or organic solvents have very small loss factors, and are thus difficult to heat with microwave irradiation.

A direct microwave heating method was developed

to measure the permittivity at high temperature when a ceramic is heated by microwaves. The sample was inserted in the cavity and directly heated by microwaves at 6 GHz. The single-mode cavity shown in Fig. 5 was used. The width of the variable iris and the plunger position were controlled to adjust the resonance and critical coupling in the cavity. Measurement of the permittivity up to 1800°C was possible with this system.⁽³²⁾ The temperature dependence of the loss factor ϵ'' is shown in Fig. 20. The values of ϵ'' and ϵ_r were measured from the iris width and the plunger position, respectively. The loss factor for Al_2O_3 increased exponentially with temperature, and the value at 1800°C was more than 100 times that at room temperature. Similarly, the loss factor for Si_3N_4 at 1700°C was approximately 60 times that at room temperature. The loss factor measured from the iris width corresponded well with that measured from the half-power width by the perturbation method. Thus, the permittivity at high temperatures can be measured easily to high accuracy with this new method.

It is very important to understand the frequency and temperature dependence of the permittivity for powders and liquids so that a suitable frequency and cavity design can be selected. These characteristic values are extremely important factors and act as

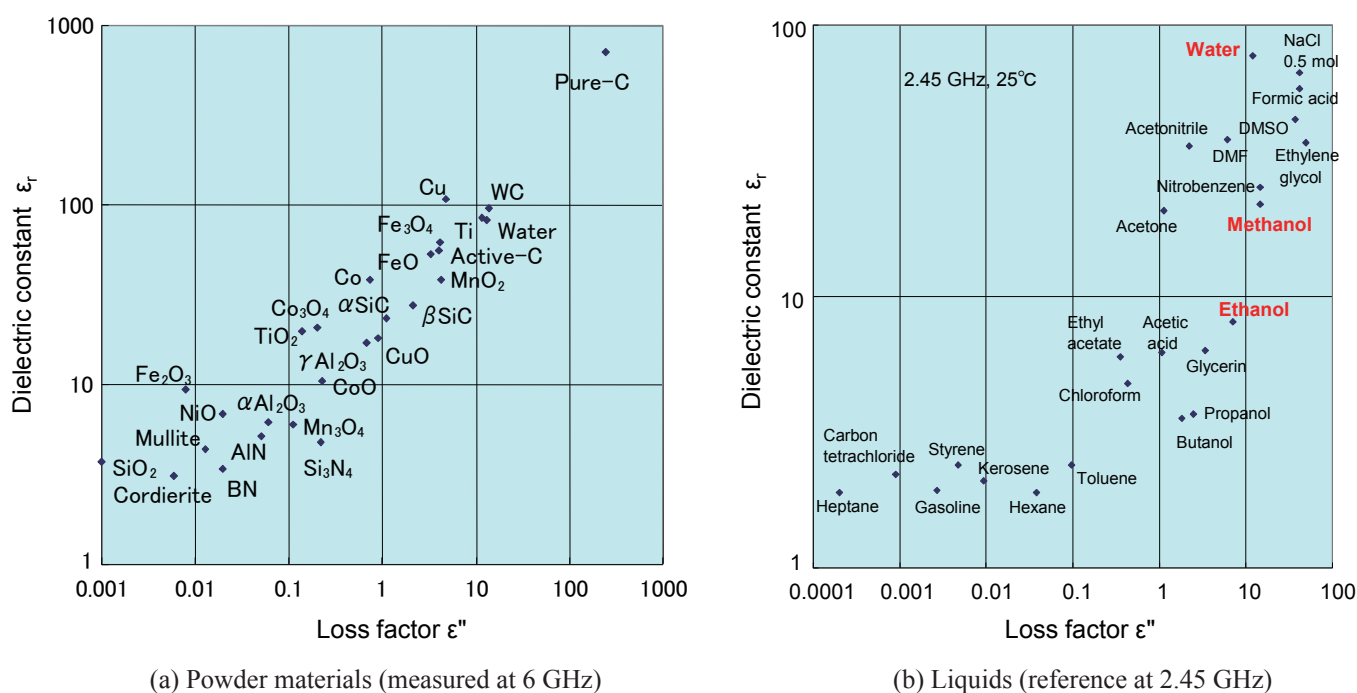


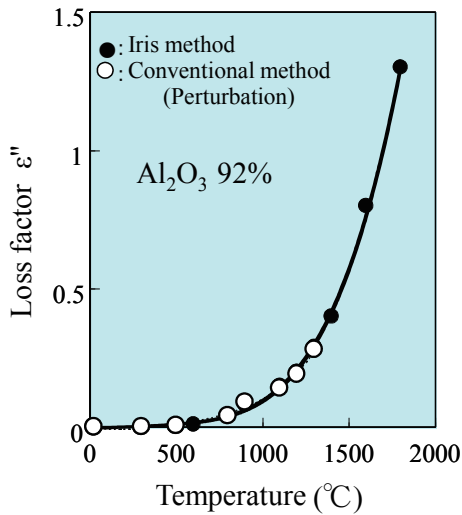
Fig. 19 Complex permittivity for various powders and liquids in microwave region.

an index to determine the advantages and economic potential of microwave processing. The S parameter by coaxial was used for powders, and the probe method was used for liquids using a vector type network analyzer.⁽²⁸⁾ The dielectric loss factors for several carbon powders and typical liquids are shown in Fig. 21. The dielectric properties of the carbon powders changed significantly with the type of material. The dielectric loss factor became large as the particle size became small and the frequency decreased. For this reason, it was considered that the dielectric of the carbon powder was influenced by space-charge polarization.⁽³³⁾ The dielectric loss factor

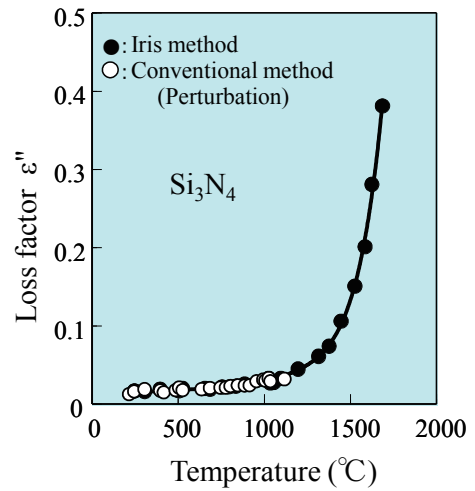
for water with an electric dipole becomes large at high frequency and low temperature, whereas ionic liquids display the opposite behavior.⁽²⁸⁾ The optimal frequency and temperature to absorb microwave energy existed in alcohol series. The peak shift of loss factor occurred due to the influence of the relaxation time of polarization.

4.2 Electromagnetic Field Distribution in the Cavity

Microwave heating employs either multi-mode or single-mode cavity resonators, expediently

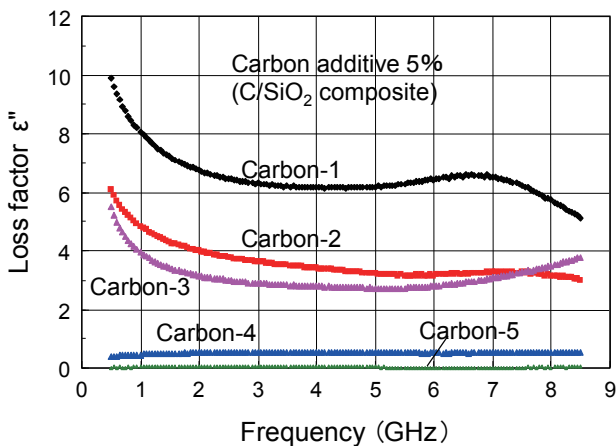


(a) Loss factor of alumina

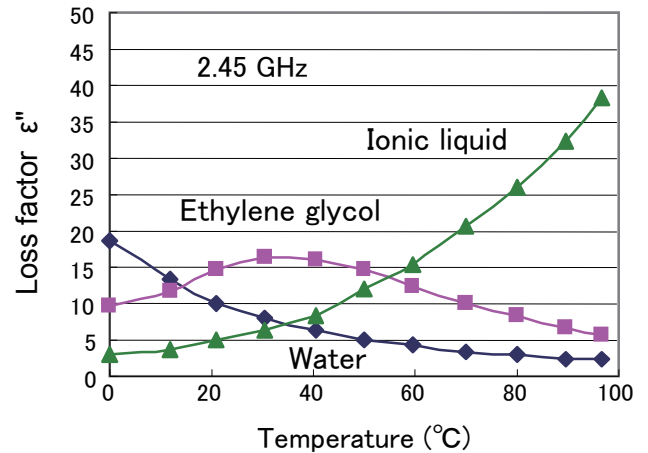


(b) Loss factor of silicon nitride

Fig. 20 Temperature dependence of dielectric properties of ceramics at 6 GHz.



(a) Loss factor of carbon powders



(b) Loss factor of typical liquids

Fig. 21 Frequency and temperature dependence of dielectric properties of powders and liquids.

classified by the number of standing waves or the electromagnetic field mode that exists in the cavity. The electromagnetic distribution in these cavities has been calculated by 3D-image analyses.⁽³⁴⁾ **Figure 22** shows the electromagnetic field distribution in a rectangular single-mode cavity (TE₁₀₃ mode) and a multi-mode cavity, i.e., a microwave oven. The electric field distribution in the single-mode cavity has three standing waves in the traveling direction of the microwaves. The mode does not change even if a sample is inserted. Dielectric and magnetic materials are rapidly heated at the positions of maximum electric and magnetic field intensity, respectively. The magnetic field distribution is opposite to that for the electric field. The locations of maximum magnetic field intensity are in the center of the cavity and on the inner wall. In contrast, the multi-mode cavity has a complex electromagnetic field distribution. The modes and maximum positions of these fields change when the sample is inserted. Therefore, it is difficult to achieve uniform and rapid heating with a multi-mode cavity. The electric field intensity E , and the magnetic field intensity H , can be expressed by the following equations:

$$E^2 = 4Q \cdot P / (\epsilon_0 \cdot \omega \cdot V), \quad H^2 = E^2 (\epsilon_0 / \mu_0), \quad (3)$$

where P is the energy introduced in the cavity, V is the cavity volume, Q is the quality factor of the unloaded (without sample) cavity. The single-mode cavity has three or four times the electric field intensity than an commercial microwave oven calculated at 2.45 GHz, i.e., the microwave absorption in a single-mode cavity is over 10 times as high as that in a multi-mode cavity.

It is necessary to evaluate the electromagnetic field distribution in the cavity to achieve uniform heating of a sample under microwave irradiation. The distribution for a multi-mode cavity is very complex compared with that for a single-mode cavity. The electromagnetic field distribution in a multi-mode type microwave oven was analyzed using the finite-difference time-domain (FDTD) method and the actual electric field intensity distribution was measured with a photoelectron field sensor.⁽³⁵⁾ **Figure 23** shows a comparison of the calculated and measured electric field distributions. Water in a 1 liter beaker was placed into the microwave oven. Five standing waves were present in the microwave oven, and the electric field intensity was reduced at the same intervals from the

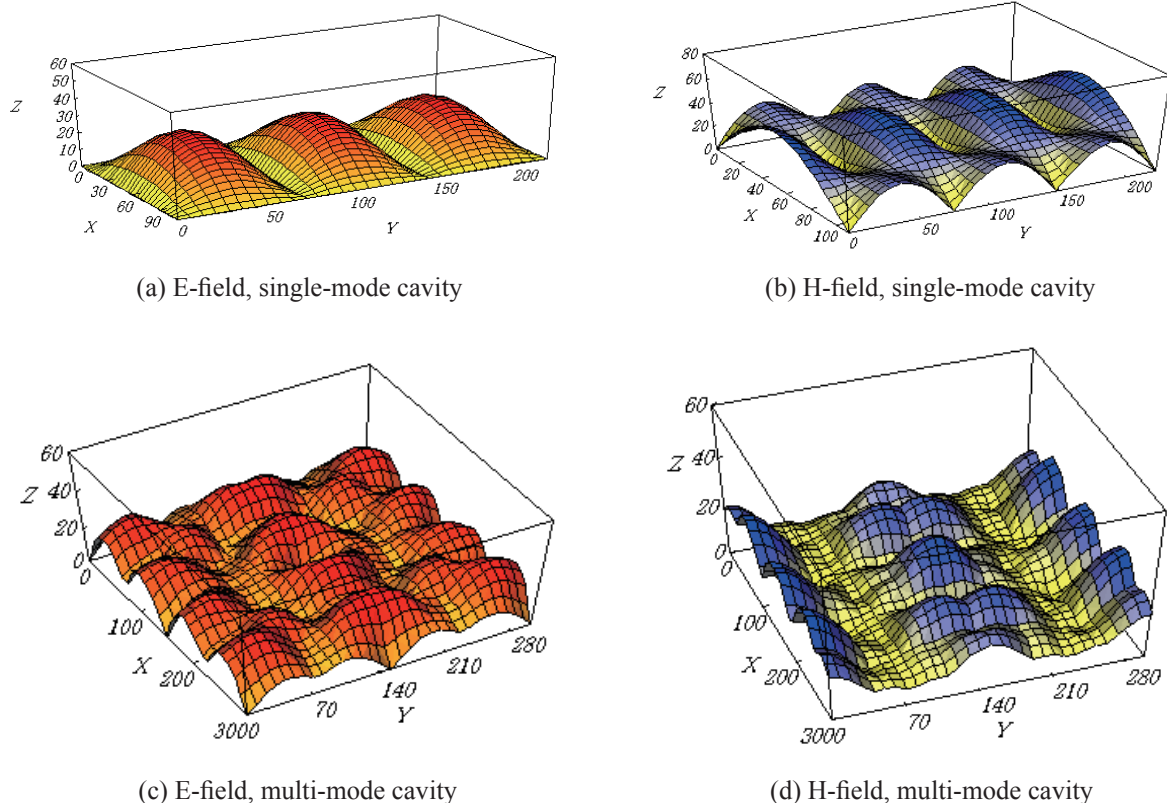


Fig. 22 Electromagnetic field distributions in single-mode and multi-mode cavities.

irradiation entrance (the right side). Furthermore, the FDTD analysis results and the measurement values corresponded well.⁽³⁶⁾ The electric field distribution in the microwave oven could thus be clarified. For that reason, to achieve uniform heating of a sample in a microwave oven, it is necessary to use sample rotation or antenna fitting to stir the electromagnetic waves.

5. Summary

The characteristics of microwave heating were presented and applications to future automobile were introduced, such as sintering of functional ceramics, hydrogen production by ethanol reforming, saccharification of cellulosic biomass, and heating of non-magnetic metal powders. Furthermore, as fundamental factors affecting microwave heating, microwave basic technologies such as the dielectric properties of materials, the electromagnetic field distribution in the cavity, and the effects of separated electric/magnetic fields were described. Ultrahigh-speed heating of over 100 °C/s was achieved using a single-mode cavity and absorbent materials. Through the selectivity of microwave heating, chemical reactions can be accelerated under a thermal non-equilibrium state. Consequently, heating at low temperature and for short times can be achieved with microwave irradiation. The following characteristics were clarified with respect to applications of the microwave process. Crystallinity was clearly promoted in the microwave sintering of ceramics. The activation

energy could be reduced in microwave reforming. A high yield was obtained during saccharification with microwave irradiation without pretreatment and expensive enzymatic requirements. The mechanism involved in microwave heating of metal powders was clarified. These innovative methods are expected to lead to breakthrough technologies for future automobile.

The reaction rate in microwave chemistry has been reported to be increased by 1-3 orders of magnitude, and synthesis without catalysts or solvents has also been possible.⁽³⁷⁻⁴⁰⁾ Microwave sintering of metal powders that had been claimed to be impossible has been shown to be possible.⁽⁴¹⁻⁴³⁾ The Japan Society of Electromagnetic Wave Energy Applications (JEMEA) was established in 2007 and this symposium has been held each year. Recently, a handbook on microwave fundamentals and applications was published in Japan.⁽⁴⁴⁾ Microwave processing has many advantages over conventional methods; however, there are problems that remain with this processing, such as the sensitivity to the powder composition and the sample size of materials. To further develop and practically use microwave heating technologies, it is necessary to improve the stability and reproducibility of these processes and to achieve a scale-up for mass production.⁽⁴⁵⁾ Further efforts are necessary to achieve mutual cooperation and collaboration with industry, academia and government, and perform basic research and development with regard to microwave technologies and applications.

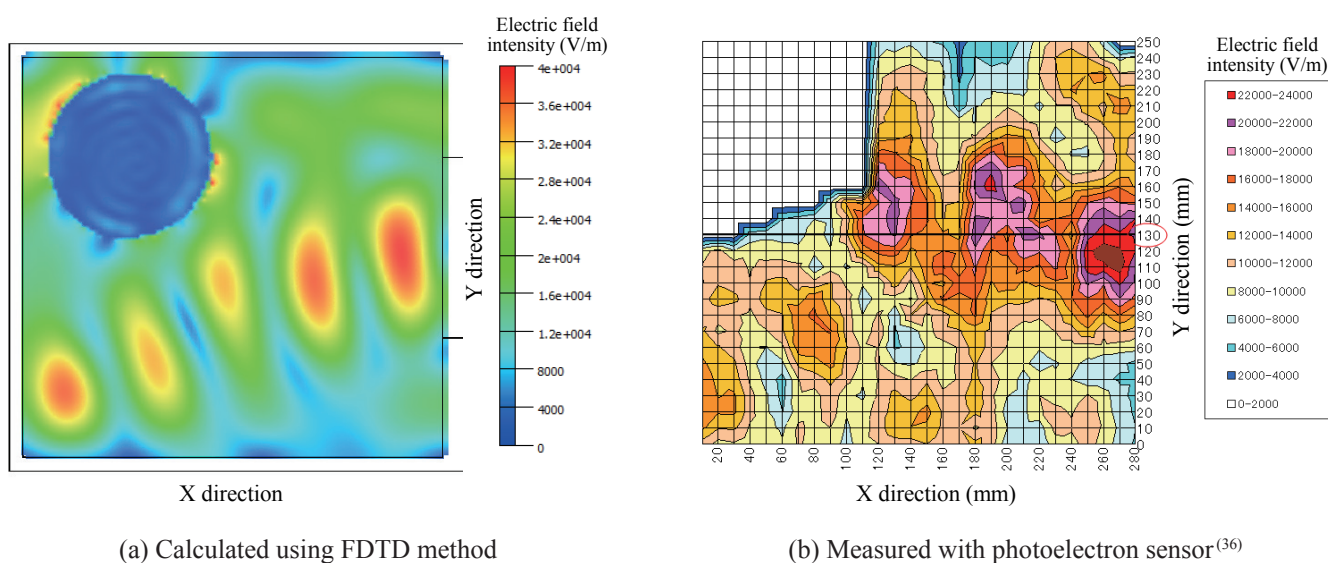


Fig. 23 Comparison of calculated and measured electric field distributions in microwave oven.

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Figs. 1 and 7(a)

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Figs. 2-3 and 9-11

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Figs. 4 and 22-23

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Figs. 5, 7(b) and 8

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Fig. 14

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Fig. 15

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Figs. 16-18

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Figs. 19 and 20

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Fig. 21

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