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Dielectric properties of MoS_2 and Pt catalysts: effects of temperature and microwave frequency

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The effects of temperature and microwave frequency on the dielectric properties of MoS_2 and Pt catalysts together with an aluminum oxide support were investigated. Dielectric constants and dielectric loss factors were measured in a temperature range of 200–800 °C by a cavity resonator technique with a cylindrical copper cavity resonating in TM_{0n0} modes ($n = 1, 2, \ldots, 5$), which corresponded to frequencies of 0.615, 1.413, 2.216, 3.020, and 3.825 GHz.

KEY WORDS: microwave; dielectric property; elevated temperature; MoS_2 catalyst; Pt catalyst; alumina support.

1. Introduction

It is well known that microwave radiations can generate heat in dielectric materials, and this has been demonstrated for a number of heterogeneous catalytic systems in which considerable enhancement in reaction rates and/or product selectivity has been reported [1–5]. Whilst the reasons for this improved performance are not fully understood, it is likely that selective heating of parts of the catalyst, together with other types of temperature variation across the catalyst bed, will be important factors [6–10]. In previous studies of the reaction of sulfur dioxide with methane and the decomposition of hydrogen sulfide using microwave heating, both over MoS_2/alumina catalysts [11,12], we have observed not only a reaction rate increase, but also a phase change in the catalysts with partial conversion of γ-alumina to α-alumina under microwave heating. This phase conversion occurs only at very high temperatures, far in excess of the measured temperature. A difference between the reaction site temperature and the bulk catalyst temperature has also been observed in other heterogeneous catalytic systems [4,5,13]. A possible explanation for this observation is that a localized thermal runaway or "hot spots" have been created in parts of the catalyst bed. This type of phenomenon can be found whenever the microwave power absorbed by the catalyst increases with temperature at a greater rate than the heat lost from the catalyst. Such thermal runaway has been confirmed by the investigation of the microwave dielectric heating behavior of supported MoS_2 and Pt catalysts [14].

In microwave heating systems, it is understood that the interaction of materials with microwaves and the heating behavior of materials by microwave irradiation are strongly dependent on their dielectric properties, along with other parameters such as the electric field and the frequency. In turn, such parameters will control the power that can be dissipated in a given material volume. One of the more important properties of such heating processes is complex permittivity (ε^*), which is expressed as

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \qquad (1)$$

where the real part, ε', is the relative dielectric constant which characterizes penetration of microwaves into the material and contributes to the stored energy of the material by polarization, and the imaginary part, ε'', is the dielectric loss factor which indicates the material's ability to convert the absorbed energy into heat. Both ε' and ε'' are frequency and temperature dependent, and a balanced combination of dielectric constant, ε', to permit adequate penetration and high dielectric loss, ε'', is required for effective microwave dielectric heating. Therefore the ratio of the dielectric loss and the dielectric constant is commonly used to measure the ability of a material to convert electromagnetic energy into heat energy at a given frequency and temperature, which is referred to as the loss tangent

$$\tan\delta = \varepsilon''/\varepsilon'. \qquad (2)$$

In the past a considerable amount of data has been obtained on the variation of ε^* with frequency and temperature for a range of materials; notable is the work of von Hippel [15]. However, whilst there have been several hundred papers published on the dielectric properties of materials, there are relatively few data which are directly

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useful to heterogeneous catalysis processes. Much of the available data refer to foodstuffs, biological materials, and ceramics at relatively low frequencies, and few data are available on the temperature variation of dielectric properties. The latter type of data are particularly necessary in order to be aware of how the heating characteristics of materials change with temperature in heterogamous catalytic systems. Numerical modeling techniques that simulate the response of catalytic reactors under microwave conditions also require detailed information about how the dielectric properties of materials change with temperature.

In this paper, we examine the effects of temperature and microwave frequency on the catalysts' dielectric properties relevant to microwave dielectric heating. The dielectric property of two $MoS_2/\gamma\text{-}Al_2O_3$ catalysts made in different ways and one $Pt/CeO_2/\gamma\text{-}Al_2O_3$ catalyst have been measured at elevated temperature ranging from 200 to 800 °C at various frequencies. The dielectric property of the $\gamma\text{-}Al_2O_3$ support and bulk MoS_2 were also measured under the same conditions. In this way it was possible to compare the contribution to the dielectric property from different catalyst species and the support.

2. Measurements of dielectric properties

2.1. Dielectric property measurement techniques

There have been a number of established methods for measuring microwave dielectric properties according to the frequency ranges used. For example, for frequencies below 0.1 GHz, bridge and *Q*-meter methods have been employed. Above this frequency the measurement has been accomplished in coaxial lines or waveguides by determining the propagation constant of the electromagnetic wave in the material. Above 10 GHz, free space methods have been used [16,17]. The choice of method is dependent upon a number of factors, of which cost, desired accuracy and loss, phase, and quantity of the materials to be studied are probably the most important.

The method based on a circular electromagnetic resonant cavity has long been employed for investigating the dielectric properties of materials in three basic ways: (a) the material completely fills its interior, (b) the material partially fills the cavity and contacts the walls at the top and bottom, and (c) the material partially fills the cavity and is held in suspension within it by a sample holder. Measurements by the first two methods at temperatures other than room temperature require cavity and material to be heated or cooled as desired. The third method, however, can be adapted to allow rapid movement of the suspended sample between a temperature regulator (*i.e.*, furnace or chiller) and the cavity. This is realized by forming sample insertion holes in the cavity walls and allowing the sample to be heated in a hot zone before returning it to the resonant cavity for measurement. Despite the increased difficulties associated with solving Maxwell's electromagnetic equations for a cavity with insertion holes and the increased uncertainties in the data obtained due to the necessity to make approximations concerning sample shape and the sample holder, this method is by far the most popular for measuring the high-temperature dielectric properties of materials in solid phase by a cavity resonator technique [18,19] and was employed in this study.

The principal arrangement of the sample and the cavity for the resonant cavity perturbation technique is shown in figure 1. For the sample dimensions and magnitude of permittivity encountered in these experiments, simple perturbation theory may be used to calculate the permittivity of the sample by the following equations [18,19]:

$$\varepsilon' = 1 + 2J_1^2(x_{0,n})\frac{V_0}{V_1}\left(\frac{f_0 - f_1}{f_0}\right) \tag{3}$$

$$\varepsilon'' = J_1^2(x_{0,n})\frac{V_0}{V_1}\left(\frac{1}{Q_0} - \frac{1}{Q_1}\right) \tag{4}$$

where V_0 and V_1 are the volumes of the cavity and sample respectively, f and Q are the resonant frequency and Q-factor of the cavity respectively where the subscripts 0 and 1 relate to the empty and loaded cavity, respectively. $J_1^2(x_{0,n})$ is the first-order Bessel function evaluated for the nth root of $J_0(x_{0,n}) = 0$. The dielectric constant ε' and loss factor ε'' can be obtained by measuring the shift of the resonant frequency and the Q-factor of the cavity, respectively.

2.2. Experimental set-up

Figure 2 shows diagrammatically the experimental set-up of the system used for the measurement of the

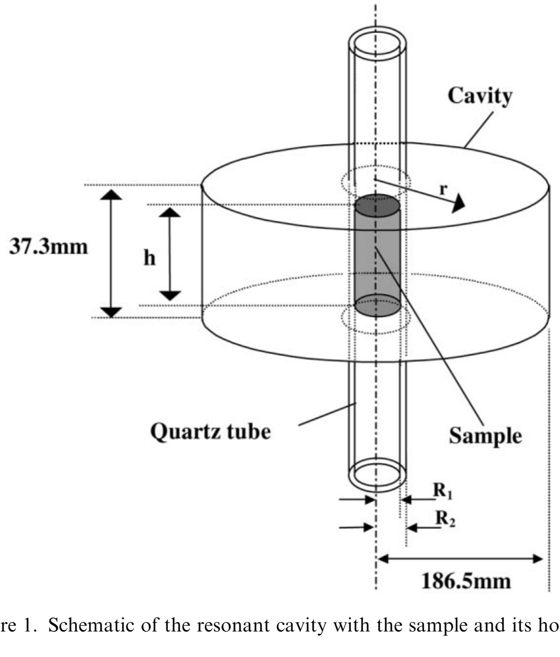

Figure 1. Schematic of the resonant cavity with the sample and its holder.

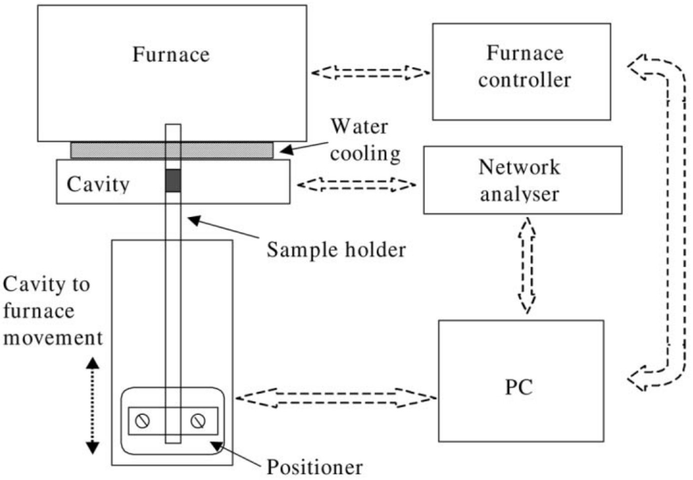

Figure 2. The experimental system for the dielectric property measurements.

dielectric property at elevated temperatures and varied frequencies.

The cylindrical resonant cavity was made of copper with internal diameter of 373.0 mm and internal height of 37.3 mm (see figure 1). A conventional tube furnace (Carbolite) capable of heating to temperatures up to 1800 °C was used to heat the sample to a desired temperature and was regulated by a P-I-D controller (Eurotherm). A motor capable of 6500 r.p.m. was controlled by the computer *via* a stepper motion controller (Unidex® 1) to move the sample holder up and down, and to ensure that the time for which the sample was removed from the furnace to the cavity for measurement was kept to a minimum (approximately 1 s). The time the sample spends outside the furnace was, in fact, a combination of traveling time and the response time of the vector network analyzer. In the standard automatic measuring operation using this motion control system the limiting factor for the time the sample spent outside the cavity was the analyzer response time. A Hewlett-Packard 8753C Vector Network Analyzer with frequencies between 0.3 and 6 GHz was used to measure the shift of the resonant frequency and the Q-factor of the cavity. The analyzer was employed in the two-port transmission mode *via* two loop aerials at 90° to each other inside the cavity. The analyzer was linked to the PC to allow automatic determination of frequency shift and quality factor for the cavity when the sample was inserted. A quartz tube and low-loss quartz wool plugs were used to hold the sample. The frequency shift and the change in Q-factor caused by the sample holder up to a furnace temperature of 1400 °C were measured prior to sample measurements. Any change in the permittivity of the sample holder with temperature can then be calibrated and separated from the measured permittivity of the sample. To prevent heat convection from the base of the furnace distorting the copper cavity during high-temperature measurement, a water-cooling system consisting of a spiral copper pipe was attached to the outer top surface of the cavity. A program written with Visual Basic was used for monitoring and controlling the furnace, the positioner and the network analyzer, and for data acquisition.

2.3. *Experimental procedure*

The sample was placed in the holding tube in an appropriate position, supported with quartz wool plugs. Argon gas was introduced into the sample holder to replace the air and then both ends of the tube were sealed under vacuum conditions. Before dielectric measurements were made the holder with sample was placed vertically and attached to the positioner and tested to make sure it could move up and down freely between the cavity and the furnace. At each temperature level, three measurements were taken when (a) the cavity was empty, (b) the empty sample tube was in the cavity, and (c) the sample with the holder were in the cavity by movement of the positioner. Then the dielectric constant and the dielectric loss could be calculated automatically according to the equations described above. The dielectric measurement was carried out in the temperature range 200–800 °C at 100 °C intervals at frequencies of 0.615, 1.413, 2.216, 3.020, and 3.825 GHz, respectively.

2.4. *Sample preparation*

MoS_2 was purchased as powder (particle size $<2\,\mu$m diameter) and activated alumina (γ-Al_2O_3) as 3.2 mm pellets from Aldrich Chemicals. The activated alumina was ground and sieved to the desired size (152–178 μm) before using.

The supported molybdenum catalysts, 30 wt% MoS_2/70 wt% γ-Al_2O_3, were prepared by impregnating the activated alumina support with ammonium heptamolybdate $[(NH_4)_6[Mo_7O_{24}]\cdot 4H_2O]$ (supplied by Alfa, Johnson Matthey plc) solution. An appropriate amount of solution, which was calculated to produce 30 wt% MoS_2

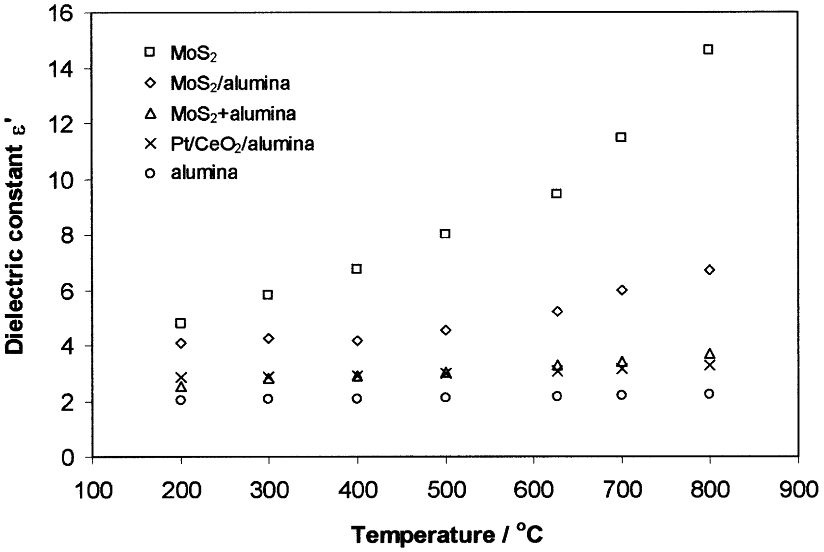

Figure 3. Dielectric constant of different samples as a function of temperature at a frequency of 2.216 GHz.

on the support, was first mixed with alumina and allowed to stand at room temperature for 6 h. The paste was then dried in a furnace with gradual increase of temperature to 105 °C over 3 h, followed by isothermal heating at 105 °C for 18 h. Once dried, the sample was calcined in a flow of air at 500 °C for 2 h. Before using, the catalyst precursor was sulfided with a mixture of H_2S/H_2 (15 mol% H_2S), both supplied by Aldrich, at a gas flow of 90 ml min^{-1}. The sulfidation temperature was 400 °C and was maintained for 2 h. All of the catalysts had alumina support particles in the size range 152–178 μm.

The mechanically mixed catalyst, 30 wt% MoS_2 + 70 wt% γ-Al_2O_3, was made by mixing MoS_2 (<2 μm) with alumina (<66 μm) first, then compressing the mixture into pellets followed by grinding and sieving to the desired size (152–178 μm).

For the supported Pt catalyst with promoters CeO_2, 8 wt% Pt/20 wt% CeO_2/γ-Al_2O_3, a two-step co-impregnation was used. The promoter CeO_2 was first impregnated onto the γ-Al_2O_3 using $Ce(NO_3)_3{\cdot}6H_2O$ (purity 99.99%, Aldrich) solution in the concentration based on the calculation. The sample was then dried at 60 °C over 18 h, then calcined in a flow of air at 500 °C for 2 h. Platinum was then impregnated onto CeO_2/γ-Al_2O_3 by mixing the $H_2PtCl_6{\cdot}6H_2O$ (Alfa, Johnson Matthey) aqueous solution, which was calculated to produce a desired content of Pt on the support, with CeO_2/γ-Al_2O_3 and allowing to stand at room temperature for 6 h. The paste was then dried in a furnace at a temperature of 60 °C over 18 h. Once dried, the sample was calcined in a flow of air at 500 °C for 2 h. Before use in reactions, the catalyst precursor was reduced with hydrogen for 1 h at a temperature of 400 °C.

The apparent densities of samples of MoS_2, γ-Al_2O_3, 30 wt% MoS_2 + 70 wt% γ-Al_2O_3, 30 wt% MoS_2/70 wt% γ-Al_2O_3 and 8 wt% Pt/20 wt% CeO_2/γ-Al_2O_3 were 990, 950, 1310, 1040, and 1430 kg m^{-3}, respectively.

3. Results and discussion

3.1. Variable temperature dielectric properties

Dielectric properties of the five samples were measured in the temperature range 200–800 °C at 100 °C intervals at a frequency of 2.216 GHz. Figure 3 shows the behavior of dielectric constant, ε', and figure 4 shows the dielectric loss factor, ε'', with temperature varying.

In general, as the temperature was increased both the dielectric constant and the dielectric loss factor increased in two stages for all the samples measured. When the temperature was in the lower range (200–600 °C), there were only small changes in both the dielectric constant and the dielectric loss factor. However, at higher temperatures (600 °C), both the dielectric constant and the dielectric loss factor increased markedly with increasing temperature, especially for samples of MoS_2 and MoS_2/γ-Al_2O_3. The critical temperature point between the two temperature ranges varied with samples, but was found to be around 500–600 °C for the samples measured. This behavior is in keeping with that of other materials measured which showed a similar critical temperature point, such as glass ceramic [20], aluminum oxide [21], and Nylon [22], and is directly relevant to the operation of microwave dielectric heating systems in the following aspects.

(a) Possibility of thermal runaway. The microwave heating equation is generally expressed as [23]

$$P_{av} = \tfrac{1}{2}\omega\varepsilon_0\varepsilon''_{eff}E^2V \qquad (5)$$

where ε_0 is the vacuum permittivity, ω is the radiation angular frequency, E is the electric field, and V is the sample volume. It can be seen that the power absorbed is proportional to the material's dielectric loss factor. If the dielectric loss factor is strongly temperature-dependent,

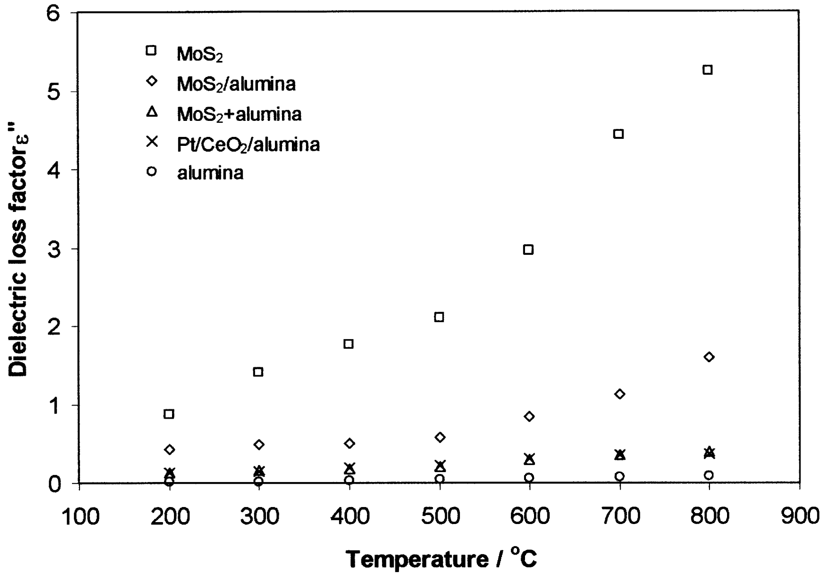

Figure 4. Dielectric loss factor of different samples as a function of temperature at a frequency of 2.216 GHz.

it can sometimes lead to two extreme situations: either the material cannot be heated to a desired temperature or a thermal runaway occurs in which the temperature continues to rise uncontrollably. In the latter case, after an initial absorption of the microwave energy, the temperature rise causes the loss factor to increase, which, in turn, results in a further temperature increase, and so on. The possibility of a thermal runaway occurring depends on the slope of the ε'' *versus* temperature response, $d\varepsilon''/dT$. For the five samples measured, $d\varepsilon''/dT$ of bulk MoS_2 is much higher than that of support γ-Al_2O_3 so the latter is very unlikely to give a thermal runaway. This conclusion is in keeping with previous studies on the microwave heating behavior of MoS_2- and Pt-supported catalysts [14].

(b) For materials with low dielectric loss, the microwave heating system may not reach the desired temperature. It is generally believed that loss factors in the range 0.01–5 would present materials suitable for microwave heating applications [23]. However, it is possible that if a reaction were initiated by other means, heating could be sustained by microwave heating at high temperatures where the loss factor is relatively higher.

(c) The increase in the dielectric loss with increasing temperature may also contribute to the formation of hot spots observed as described previously for heterogeneous catalysts [11–13].

According to Kenkre *et al.* [24] there are two types of microwave-absorbing entities present in ceramic materials. The first, referred to as type A, are localized absorbers that contribute a temperature-independent term to $\varepsilon''_{\mathrm{eff}}$. The second, referred to as type M, are interstitial atoms or vacancies, which are localized in shallow potential wells at low temperatures but become mobile at higher temperatures. The strong increase in $\varepsilon''_{\mathrm{eff}}$ with temperature rising is thought to be caused by the increased mobility of these type M absorbers. If the activation energy required to cause these entities to become mobile is E_a, then the fraction $f(T)$ that are mobile at a given temperature will be given by the equation

$$f(T) = \exp\left(-\frac{E_a}{RT}\right)\left[\exp\left(-\frac{E_a}{RT}\right)+1\right]^{-1}. \tag{6}$$

Following Kenkre *et al.*, the variation of the energy dissipation factor, or loss tangent with temperature, can then be written as

$$\tan\delta = k_A + k_M \exp\left(-\frac{E_a}{RT}\right) \tag{7}$$

where the constants k_A and k_M are measures of the contribution made by microwave absorbers bound in deep and shallow potential wells respectively. It follows from equation (7) that $\varepsilon''_{\mathrm{eff}}$ will rise approximately exponentially with temperature.

This model has been tested against the measurement data. Figure 5 shows a plot of $\ln(\tan\delta - k_A)$ *versus* $1000/T$ for the MoS_2/γ-Al_2O_3 catalyst where $k_A = 0.102$. It shows that this model accurately describes the data. Equally good plots were obtained for other samples measured and the values found for k_A, k_M and E_a are summarized in table 1. The activation energy obtained with γ-Al_2O_3 is higher than the value of 21.6 kJ mol^{-1} calculated by Kenkre *et al.* [24], likely due to the difference in physical structures of the alumina used. The activation energies measured with MoS_2 and Pt/CeO_2 catalysts are in reasonable agreement with literature values in a range of 20–30 kJ mol^{-1} [25,26].

It was found that the activated energy obtained with pure MoS_2 was the lowest and that with pure γ-Al_2O_3 support was the highest, while the values with γ-Al_2O_3-supported MoS_2 and Pt catalysts fell within the observed

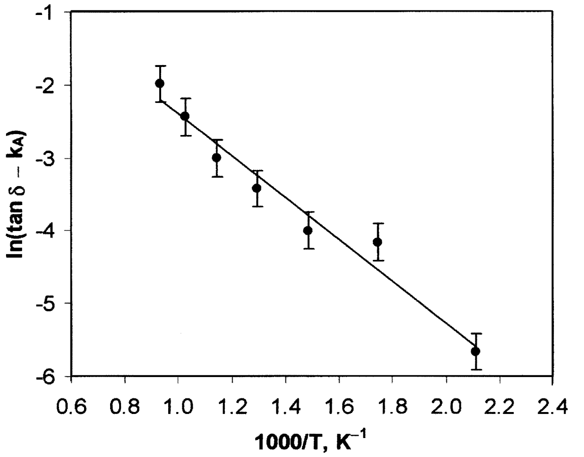

Figure 5. Plot of $\ln(\tan\delta - k_A)$ *versus* $1000/T$ for impregnated catalyst $MoS_2/\gamma\text{-}Al_2O_3$, $k_A = 0.102$.

range. This shows that the mobile absorbing entities on both impregnated and mixed catalysts are associated with neither the support material nor the catalyst species in a direct way but must be located at the catalyst/support interface. If the bulk properties of MoS_2 were primarily responsible for the temperature-dependent microwave absorption, then the activated energies for the two MoS_2 catalysts would have a similar value to that of pure MoS_2. On the other hand, the two MoS_2 catalysts made *via* different methods, *i.e.*, impregnating and mechanically mixing, should differ in the catalyst/support interface and give different activated energies. It was also interesting to note that the activated energy of $Pt/CeO_2/\gamma\text{-}Al_2O_3$ catalyst was close to that of $MoS_2/\gamma\text{-}Al_2O_3$ catalyst, even though it is generally thought that the supported platinum particles tend to behave differently from dielectric materials. A possible explanation is that the $CeO_2/\gamma\text{-}Al_2O_3$ interface effect was similar to that of $MoS_2/\gamma\text{-}Al_2O_3$ where the loading of CeO_2, 20 wt%, was considerable bigger that that of Pt, 8 wt%.

Table 1
Values of the constants contained in equation (7) for all the samples measured.

Catalyst	k_A	k_M	E_a (kJ mol^{-1})
MoS_2	0.122	0.71	9.3
MoS_2/alumina	0.102	1.48	23.5
MoS_2+alumina	0.055	6.24	39.7
Pt/CeO_2/alumina	0.046	1.03	22.8
Alumina	0.010	7.87	45.7

3.2. Frequency dependence of dielectric properties

To avoid interfering with radar and telecommunication frequencies, most of the industrial and domestic applications of microwaves are authorized to operate at frequencies within given bands that are carefully selected, and the frequencies of 0.915 and 2.45 GHz are mostly used. Therefore, the measurement of the dielectric property is often carried out around these frequencies.

Figures 6–10 show the dielectric constants and loss factors measured at varied frequencies of 0.615, 1.413, 2.216, 3.020, and 3.825 GHz in the temperature range 200 to 800 °C for the five samples, respectively.

It was observed that all the samples showed the same generic frequency dependence for the dielectric constant ε'. When the frequency was increased the dielectric constant ε' was found to decrease. It was also observed that this decrease in ε became more significant with increasing temperature, while there was only a small change in ε' when the temperature was in the lower range (<500 °C).

However, the frequency dependence of the dielectric loss factor ε'' varied with the material measured. For the pure $\gamma\text{-}Al_2O_3$ support and impregnated $MoS_2/\gamma\text{-}Al_2O_3$ catalyst, the dielectric loss factor ε'' decreased with increasing frequency although they were overall low. In contrast, for pure MoS_2 and mechanically mixed $MoS_2 + \gamma\text{-}Al_2O_3$ catalyst, the dielectric loss ε'' increased with increasing frequency. In general, this

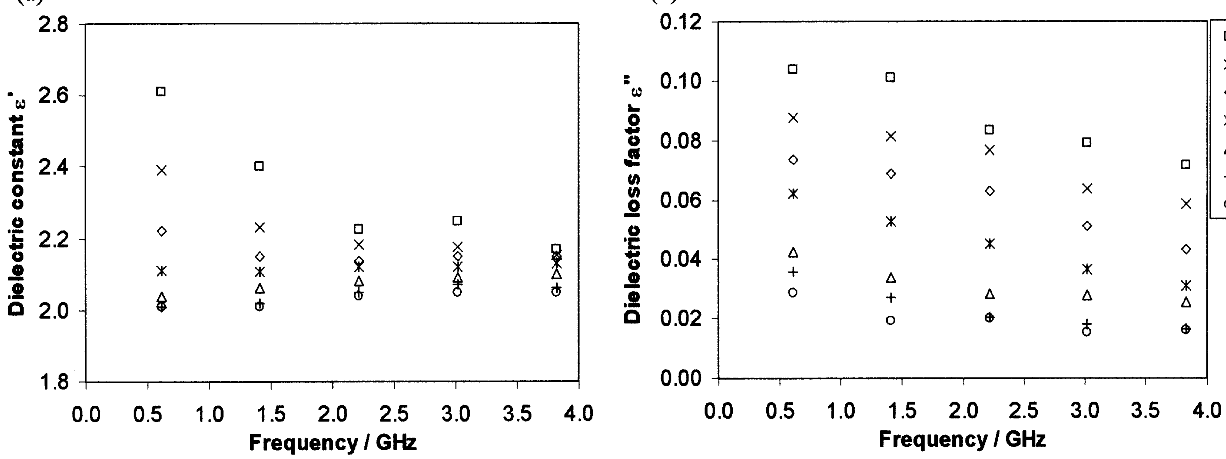

Figure 6. Dielectric properties of $\gamma\text{-}Al_2O_3$ *versus* microwave frequency at different temperatures.

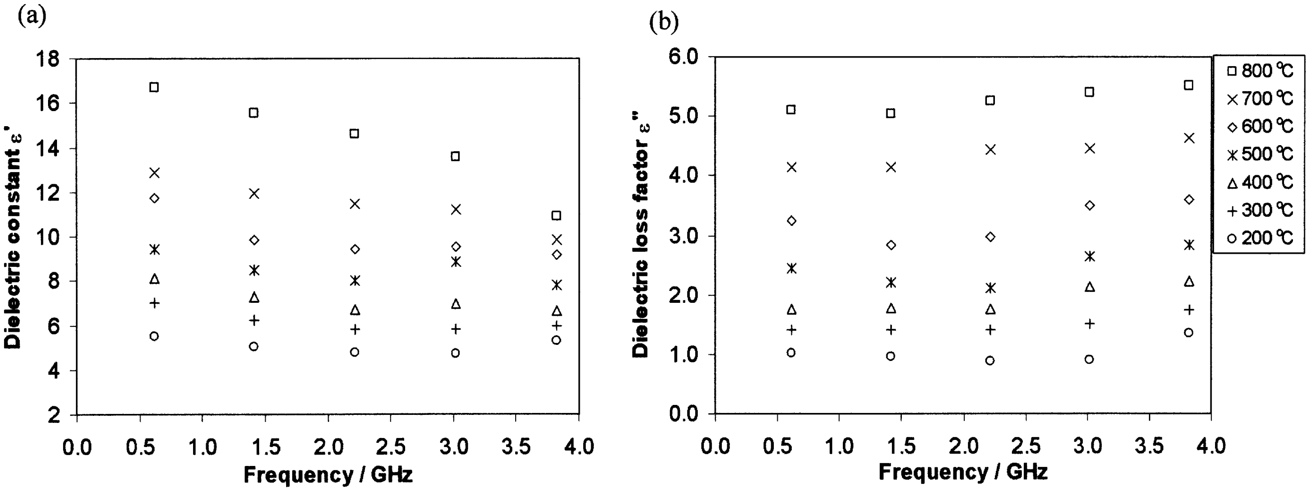

Figure 7. Dielectric properties of MoS_2 *versus* microwave frequency at different temperatures.

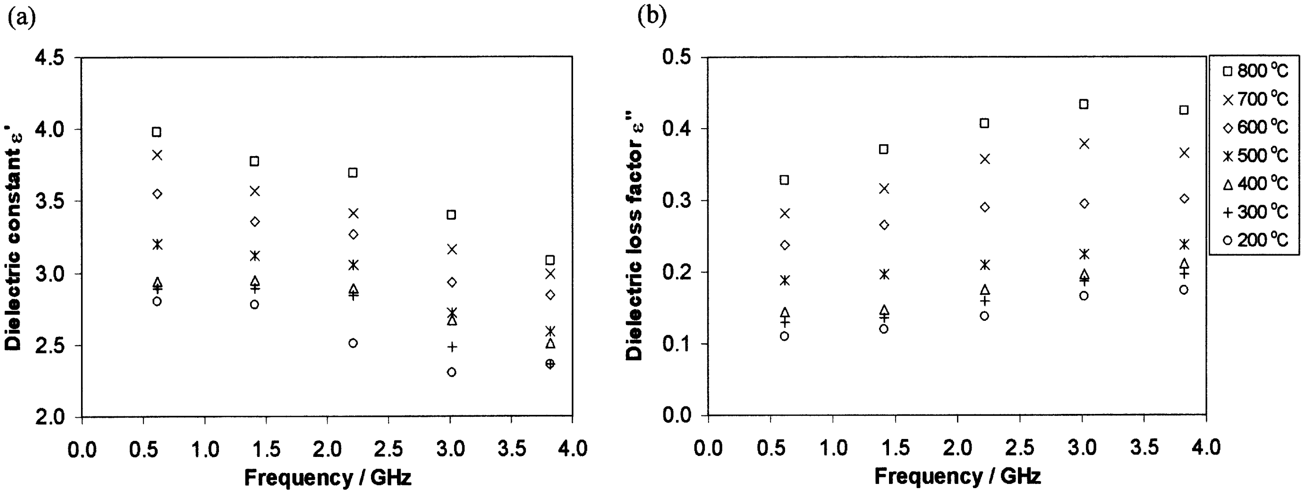

Figure 8. Dielectric properties of mechanically mixed catalyst $MoS_2 + \gamma\text{-}Al_2O_3$ *versus* microwave frequency at different temperatures.

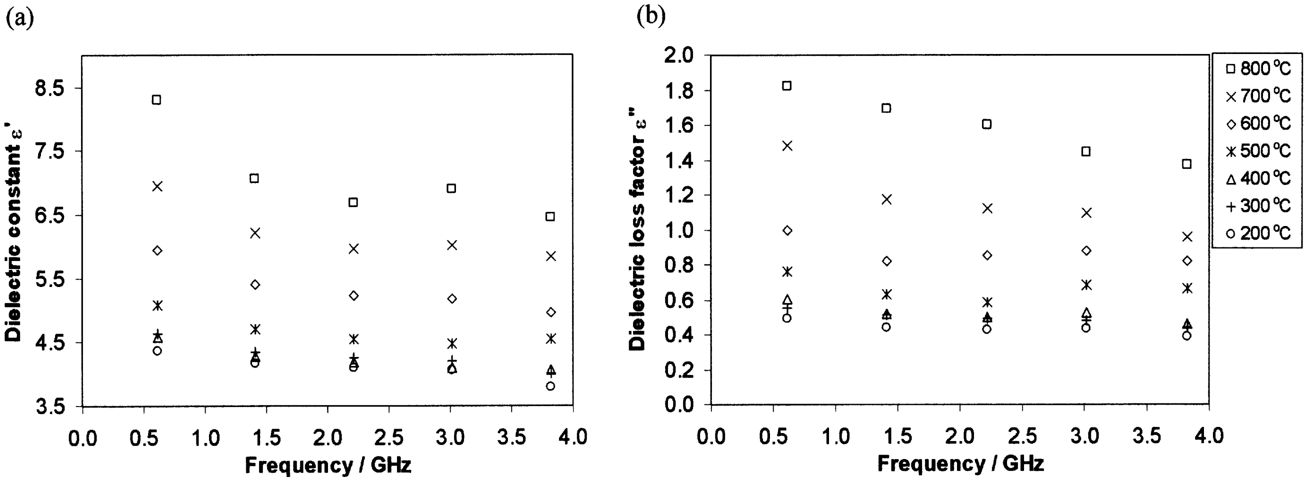

Figure 9. Dielectric properties of impregnated catalyst $MoS_2/\gamma\text{-}Al_2O_3$ *versus* microwave frequency at different temperatures.

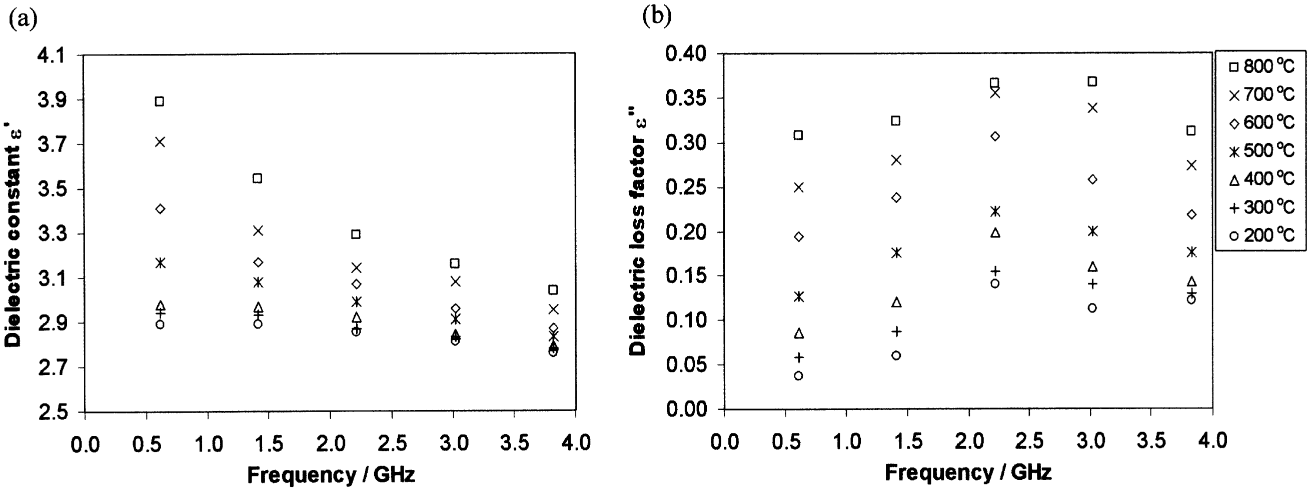

Figure 10. Dielectric properties of impregnated catalyst $Pt/CeO_2/\gamma\text{-}Al_2O_3$ *versus* microwave frequency at different temperatures.

decrease or increase in ε'' was more pronounced as the temperature was increased. The variation of the dielectric loss factor ε'' of $Pt/CeO_2/\gamma\text{-}Al_2O_3$ catalyst with frequency, however, differed from other samples and showed a maximum at about 2.5 GHz.

It is interesting to note that the opposite trend of ε'' with frequency was observed with the supported and mechanically mixed catalysts of MoS_2 in $\gamma\text{-}Al_2O_3$ with the same contents (30 wt% MoS_2). The mechanically mixed catalyst $MoS_2 + \gamma\text{-}Al_2O_3$ showed a similar trend to pure MoS_2, while impregnated catalyst $MoS_2/\gamma\text{-}Al_2O_3$ followed the trend for pure $\gamma\text{-}Al_2O_3$. This result is likely to be due to the physical structure difference in the two catalysts, as MoS_2 was evenly dispersed over the surface of the $\gamma\text{-}Al_2O_3$ support while the $\gamma\text{-}Al_2O_3$ support played a volumetric role in the mechanical mixture.

The dielectric loss arising from interfacial polarization in a system composed of a conducting phase dispersed in a loss-free medium has been examined by a number of authors [9,27–29], and it was found that the dielectric loss factor was related to conductivity, σ, and radiation angular frequency, ω, in terms of σ/ω. For the $Pt/CeO_2/\gamma\text{-}Al_2O_3$ catalyst, the supported platinum tends to form small conducting particles where the conductivity is also frequency-dependent. Therefore, the variation of the loss factor of the $Pt/CeO_2/\gamma\text{-}Al_2O_3$ catalyst with frequency differed from other dielectrics measured.

4. Conclusions

Using a cylindrical copper cavity resonating in TM_{0n0} modes ($n = 1, 2, \ldots, 5$), the dielectric property of two $MoS_2/\gamma\text{-}Al_2O_3$ catalysts produced in different ways and one $Pt/CeO_2/\gamma\text{-}Al_2O_3$ catalyst have been measured at elevated temperature ranging from 200 to 800 °C at frequencies of 0.615, 1.413, 2.216, 3.020, and 3.825 GHz. The dielectric property of the $\gamma\text{-}Al_2O_3$ support and bulk MoS_2 were also measured under the same conditions. The measurements showed that both the dielectric constant and the dielectric loss increased with increasing temperature. Both dielectric constant and loss factor of bulk MoS_2 were the highest and those of support $\gamma\text{-}Al_2O_3$ were the lowest. For all the samples measured, the energy dissipation factor, or loss tangent, was found to increase with temperature according to the relationship

$$\tan\delta = k_A + k_M \exp\left(-\frac{E_a}{RT}\right).$$

It was demonstrated that all the samples showed the same generic frequency dependence for the dielectric constant ε' that decreased with frequency increasing in the measured frequency range. However, the frequency dependence of the dielectric loss factor ε'' varied with the material measured, mainly due to the difference in the physical structure and interface effects between the samples.

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