

Kinetics of the Microwave-Heated CO Oxidation Reaction over Alumina-Supported Pd and Pt Catalysts

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The kinetics of the CO oxidation reaction on Pt/Al₂O₃ as well as Pd/Al₂O₃ have been measured in a packed bed reactor heated by microwave energy. The reaction rate was also measured in an identical reactor that was heated via conventional means. Several methods of temperature measurement were explored for the determination of catalyst temperature in the microwave heated reactor. Insertion of a thermocouple after the microwaves have been switched off provided the best method for temperature measurement in this study. However, large-scale temperature gradients must be eliminated before this method can provide accurate temperature measurements. After suitable modifications in our packed bed reactor to eliminate nonisothermality, we found that the rate of reaction and the reaction kinetics (order of reaction) for the microwave heated CO oxidation reaction were comparable to those for the conventionally heated reactor. The CO oxidation reaction therefore serves as an *in situ* probe of metal surface temperature. These observations suggest that the Pt and Pd crystallites in a supported catalyst are not significantly hotter than the alumina support in a microwave heated reactor.

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INTRODUCTION

The benefits of microwave heating have been known for some time and have been exploited by organic chemists for liquid phase chemical reactions carried out in the batch mode (1–3). Microwave batch reactors that are transparent to microwaves but can sustain high internal pressures have also been designed (4) and result in faster reaction times due to rapid heating. The potential of microwave heating for heterogeneous catalysis prompted the Electric Power Research Institute (EPRI) and the National Science Foundation (NSF) to organize a workshop on microwave effects in April 1993 (5). At this workshop, there were several reports of how microwave heating could enhance the rates as well as selectivity of chemical reactions. For example, Cha reported high removal rates (98%) of SO₂ and NO_x in a

microwave heated char-bed (6) and rapid devolatilization of char and coal in a microwave heated reactor (7). There are several other reports of microwave-assisted heterogeneous catalysis in the literature (8–10).

The benefits derived from microwave heating of catalytic systems appear to fall into one of two categories: rapid heating and temperature gradients within the reactor. In the liquid-phase batch mode experiments (1–3), rapid heating can be attributed to the mode of energy transfer. In a conventionally heated system, heat must be transferred from the source and into the reaction vessel via conduction and convection. In a microwave-heated vessel, the microwave energy is converted to heat within the vessel through coupling with the polar solvents, bypassing conventional heat transfer mechanisms. Similar benefits are seen also in heterogeneous gas-phase processes: Cha (6, 7) has shown that rapid heating occurs in a microwave-heated char bed. Also, Wan and Koch (8) show kinetic plots where the product formation in the ammonia synthesis of HCN reaches a quasi-steady state in a matter of seconds. In these heterogeneous experiments, microwave energy is dissipated directly into the solid catalyst. As with the liquid phase reactions, microwave heating bypasses the conventional convective and conductive heat transfer modes with the result that heating is rapid.

In addition to rapid heating, microwave heated heterogeneous systems may provide temperature gradients within the catalyst bed, with potentially beneficial effects. The work by Cha *et al.* (6, 7) suggested that large temperature gradients may exist between the char particles and the gas stream in the packed bed with the result that the decomposition of NO_x and SO₂ occurs at a lower gas temperature. Ioffe *et al.* (9) also suggested that temperature gradients may have been responsible for their observed selectivity of >90% for acetylene formation from methane in a microwave-heated activated carbon bed. The authors suggest that the highly nonisothermal nature of the packed bed might allow reaction intermediates formed on the surface to desorb into a relatively cool gas stream where it

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follows a different reaction pathway than in an isothermal reactor. A higher selectivity towards acetylene formation was also reported by Chen *et al.* (10) during oxidative coupling of methane in a microwave heated reactor. The nonisothermal nature of the microwave heated catalyst and the lower temperature during reaction was thought to account for the improved selectivity.

Temperature gradients within a packed bed have been found to be beneficial in several applications: microwave heated infiltration (11, 12), catalyst preparation (13, 14), drying (15), and sintering (16). Modeling of power/electric field density in heterogeneous systems has also been performed (17–19). Based on their heat transfer model for a microwave heated fluidized bed, Rousy *et al.* (18) suggest, contrary to comments in (6, 7, 9, 10), that the temperature of the catalyst is identical to that of the gas phase. A very complete review of the literature was done by Peterson (20).

In a microwave heated catalyst bed, temperature gradients could occur on two length scales. Large-scale non-isothermality can be measured via a thermal imaging camera or an optical pyrometer. However, temperature gradients between the metal particles and the gas phase or support are difficult to measure. It is possible that some of the differences between microwave and conventionally heated reactors reported in the literature may be a result of temperature nonuniformities and errors in temperature measurement. Therefore, the primary objective of the present study was to perform accurate temperature measurements in a microwave heated reactor. The temperature dependence of the CO oxidation reaction over Pd and Pt on γ -Al₂O₃ was studied in an identical reactor heated via microwave as well as conventional heating. This reaction was chosen because the reaction kinetics are well known (21–23). Our results yield insight into the nature of the thermal gradients in a packed bed reactor and the kinetic behavior of the microwave-heated CO oxidation reaction.

EXPERIMENTAL

The CO oxidation reaction was studied over Pt and Pd/ γ -Al₂O₃ in a specially constructed microwave heated reaction system. The catalysts were obtained from commercial vendors: 5 wt% Pd/ γ -Al₂O₃ Escat 14 (Engelhard) and 1 wt% Pt/ γ -Al₂O₃ (300 m²/g) (Stk No. 11797, lot E31B13, AESAR). Figure 1 is a schematic of the experimental system which consists of two branches which are identical in every respect except for the heating source. Each branch consists of a stand-alone reactor, one of which is heated using microwaves and the other is heated conventionally. The reactors were constructed using 8 mm diameter quartz tubing. The thermal reactor system was heated with a digitally controlled 1" resistive tube furnace. The microwave reactor was heated by a magnetron based system operated on a continuous basis at 915 MHz. The power incident on

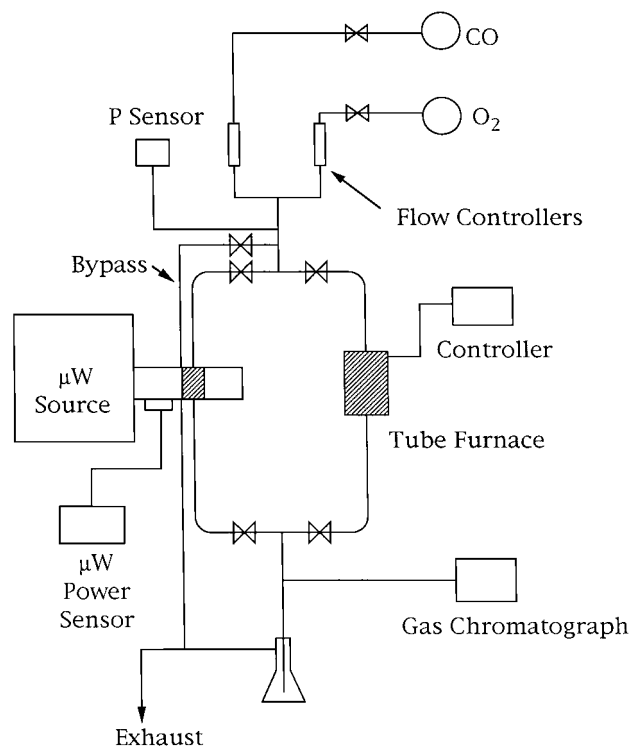


FIG. 1. Schematic of the reactor system containing two identical reactors, one heated by microwaves and the other heated via a conventional clam-shell furnace.

the microwave cavity was 850 to 1000 W and was delivered to a resonant applicator via a rectangular waveguide in the TE₁₀ mode. The cavity was structured so that only a small fraction of the incident power was absorbed by the catalyst bed, the remainder being dumped into a water load. The reactor tube, containing the catalyst, was inserted through the rectangular resonant applicator parallel to the direction of the electric field. Product analysis was performed using a Microsensor Technology M200 gas chromatograph using a 4-m PoraPlot U column. Reactant ratios were fixed by mass flow controllers, and the pressure was regulated by a bypass valve and monitored with a capacitance manometer.

The reactants were mixed in a stoichiometric ratio. The flow rate for CO was 2 cc/s and 1 cc/s for O₂, and the reactor pressure was fixed at 1000 Torr. The dependency on CO partial pressure was found by using an excess of oxygen. The oxygen flow rate was 30 cc/s and the CO flow rate was varied from 0.5–3 cc/s, or a CO partial pressure range of 35–95 Torr. The total reactor pressure was again held constant at 1000 Torr and the reactor temperature was 408 K.

An infrared thermal imaging camera was used, primarily to determine the temperature distributions in the packed bed. The spectral response of the system was such that the quartz reactor tube was translucent. Hence, the measured temperature was an average of that of the outer surface of the quartz tube and the catalyst inside (20). In view of the

radial temperature gradient within the packed bed, we felt the method would be unsuitable for accurate measurement of catalyst bed temperature. Hence, thermal imaging was abandoned in favor of the thermocouple insertion technique. However, the thermal imaging system did provide information on the spatial variation in temperature in the axial direction.

Temperature was measured in the microwave system by inserting a thermocouple into the bed after the microwave power was turned off. The temperature decay curve was recorded on a strip chart recorder. The temperature versus time curve was linearized by taking the natural log of the data and extrapolated to zero time to determine operating bed temperature. Since insertion of the thermocouple into the bed caused an initial cooling which was too rapid to appear on the strip chart curve, a correction factor was necessary. Therefore the procedure was repeated with a conventional tube furnace. The digitally controlled tube furnace was allowed to reach a known temperature, the furnace power was terminated and the thermocouple inserted while the temperature was recorded as a function of time. Figure 2 shows actual reactor temperature versus the extrapolated temperature after thermocouple insertion. This data was then used to apply a correction factor for the temperature measurement during microwave heating. Further details of the correction procedure and the error analysis are presented in the Appendix.

Initial experiments used an integral packed-bed configuration using 2 g of 1% Pt/ γ -Al₂O₃ as shown in Fig. 3. The configuration was such that the thermocouple came into contact with the top of the packed bed after insertion. As

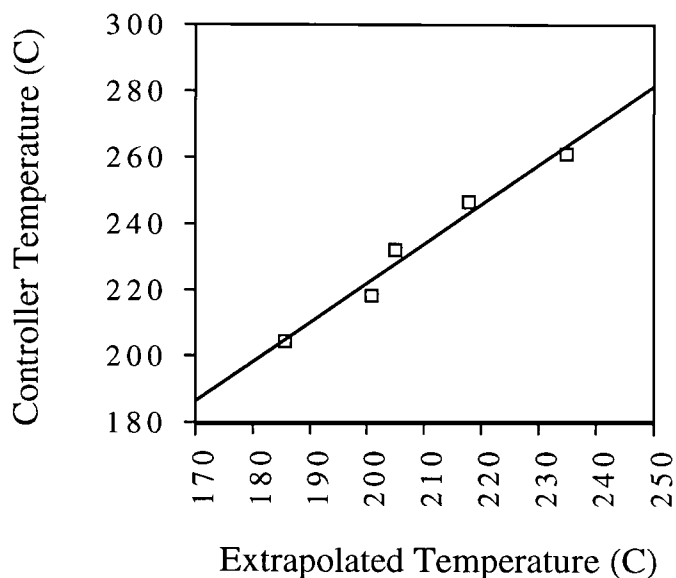


FIG. 2. Correlation between reactor temperature measured by the furnace controller and the temperature extrapolated from a thermocouple inserted into the furnace after heating had been switched off.

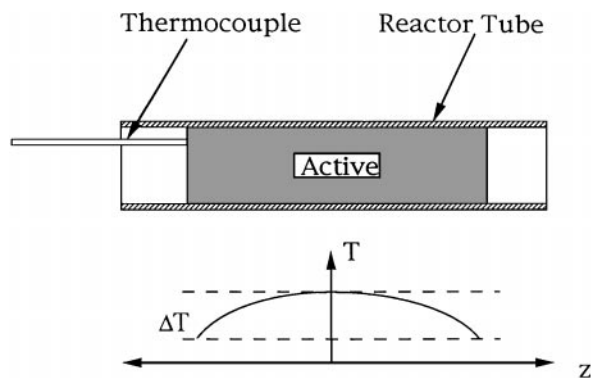


FIG. 3. Schematic of an integral-type packed bed reactor and the expected temperature profile when heated by microwaves.

we gained a better understanding of the thermal gradients in the microwave heated reactor, the packed bed configuration was modified. The evolution of the reactor configuration is described in the results section. Subsequent experiments were performed with a differential bed configuration using 5% Pd/Al₂O₃ as the catalyst.

RESULTS

Kinetics in an Integral Bed Reactor

We first present results obtained early in the experimental development, and how these results led to the improper conclusion that a microwave-induced rate enhancement was occurring. Figure 4 shows an Arrhenius plot obtained from the integral reactor structure which used 2 g of 1% Pt/Al₂O₃. The thermocouple extrapolation technique was

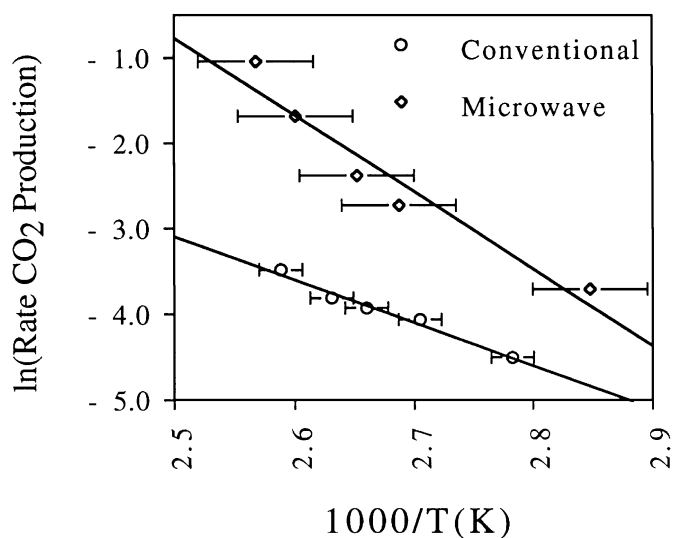


FIG. 4. Arrhenius plot for CO oxidation obtained using the configuration shown in Fig. 3. The increased reactivity under microwave heating can be attributed to errors in temperature measurement inherent in the experimental configuration of Fig. 3. Reaction rate is in units of $\mu\text{mol/s/g}$.

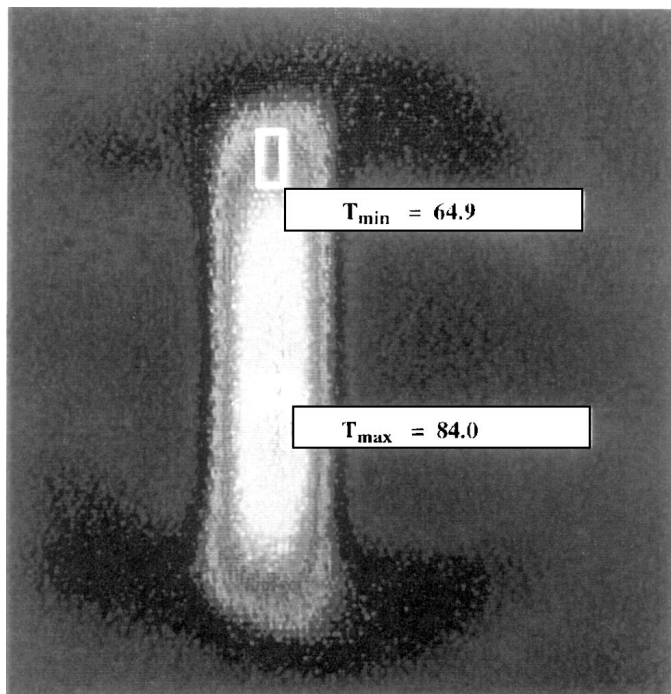


FIG. 5. Thermal (infrared) image of microwave heated integral bed. Temperatures shown are the average temperatures of the areas outlined by the boxes.

used for this experiment and the probe was inserted such that it was in contact with the top of the packed bed as shown in Fig. 3. In addition to the thermocouple, an infrared camera was used to obtain information about the temperature distributions in the packed bed. A typical infrared image from the microwave heated reactor is shown in Fig. 5. The observations indicated that temperature gradients were approximately 20 K, such that the thermocouple was recording temperature in the coolest region while most of the reaction was occurring on surfaces which were significantly hotter. The nonisothermal nature of this packed bed resulted in an apparent rate enhancement and altered activation energy and pre-exponential factor, as shown in Fig. 4.

Kinetics in a Differential Bed Reactor

Figure 6 shows a modified experimental design and the expected temperature profile. In this experimental configuration, the catalyst bed was sandwiched between two 1-g masses of inactive α -alumina support. The catalyst bed contained 3 mg of active catalyst (5 wt% Pd/ γ - Al_2O_3) diluted in approximately 50 mg of γ -alumina support to achieve radial uniformity of catalyst distribution across the packed bed. The packed bed was insulated on the outside with microwave transparent sapphire wool. In addition, a "guide tube" was added such that the thermocouple was inserted

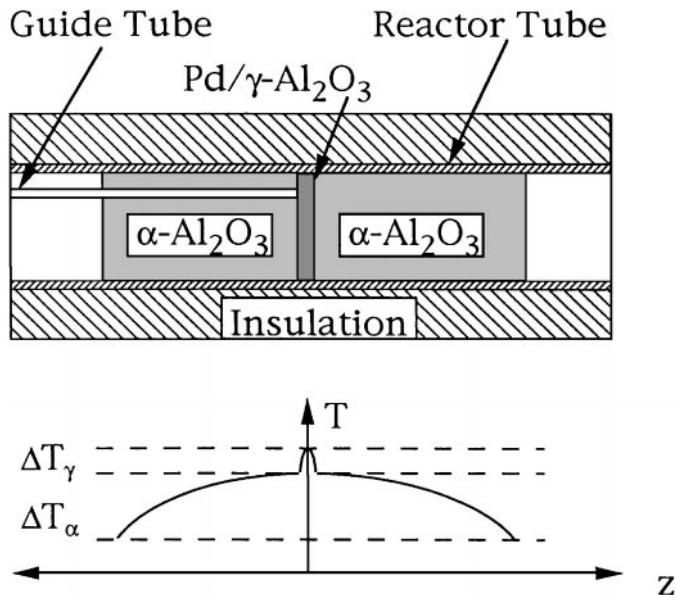


FIG. 6. Schematic of a differential packed bed reactor and the expected temperature profile when heated by microwaves.

through the tube (see Fig. 6) until it came to rest in the hottest (active) part of the bed.

Figure 7 shows an Arrhenius plot obtained from this differential-bed configuration. The plot clearly shows an apparent rate enhancement for the microwave heated reactor. As was shown before with the integral bed, the apparent rate enhancement could be caused by our inability to measure the reactor temperature properly. A blank

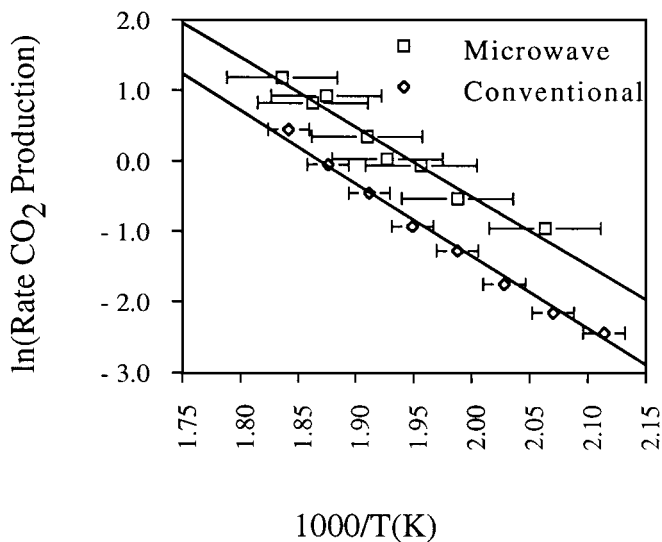


FIG. 7. Arrhenius plot for CO oxidation obtained using the configuration shown in Fig. 6. The apparent rate enhancement resulted from thermal gradients caused by differences in microwave coupling within the γ and α alumina portions of the differential bed. Reaction rate is in units of $\mu\text{mol/s/g}$.

experiment demonstrated that γ - Al_2O_3 absorbs microwave energy more efficiently than does α - Al_2O_3 . Hence, the true temperature in the catalyst bed, which is composed of γ - Al_2O_3 , may be greater than the temperature of the α -alumina which is used to sandwich the bed. The larger mass of the α -alumina (2 g) versus that of the catalyst bed (0.05 g) would make it likely that the temperature recorded by the thermocouple is lower than the actual temperature of the catalyst bed.

Kinetics in a Modified Differential Bed

For the final set of experiments which revealed the true kinetics, the differential bed was modified as described below. In this configuration, identical γ - Al_2O_3 material was used as the support as well as the insulating material surrounding the catalyst. Figure 8 shows a schematic and the expected temperature profile. An Arrhenius plot is shown in Fig. 9 for the oxidation of CO over Pd/ γ - Al_2O_3 in the modified differential bed. The reaction rates were marginally greater, for microwave versus conventional heating, but within the margin of experimental error. The slopes of the curves yield an activation energy of 12.5 kcal/mol, which agrees with the results of Choi *et al.* (23) for this temperature range. The error bars on the microwave curve include the error in the original measurement, and the propagated error from the correction procedure. The details of the error analysis have been presented in the Appendix. Also, as shown in Fig. 10, the orders of reaction in CO are nearly identical with the best fit line slopes of -0.59 and -0.64 for conventional and microwave heating.

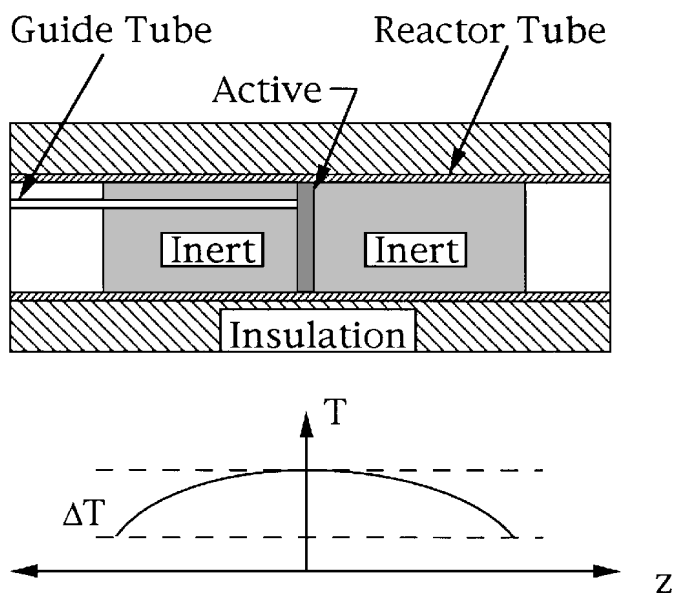


FIG. 8. Schematic of the modified differential bed configuration with the expected temperature profile.

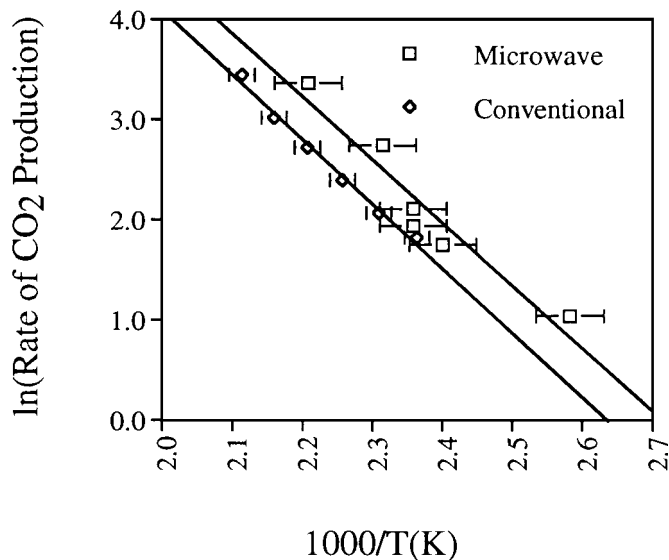


FIG. 9. Arrhenius plot for CO oxidation in the reactor configuration shown in Fig. 8. The marginal increase in rates for the microwave heated catalyst arise due to errors in temperature measurement, as discussed in the text. Reaction rate is in units of $\mu\text{mol/s/g}$.

DISCUSSION

An important question in the study of microwave catalysis is whether or not the observed effects (altered reactivity and/or selectivity) can be explained by errors in temperature measurement (20). This would be the case if the observed effects are seen also with conventionally heated

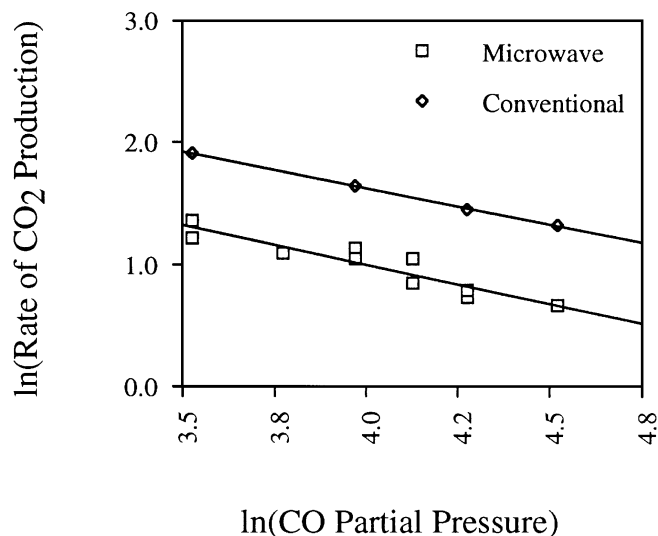


FIG. 10. Effect of CO partial pressure on CO oxidation rates for microwave and conventionally heated reactors. The order of reaction is 0.59 and 0.64 for the conventional and microwave reactors, respectively. The reaction temperature for the conventional reactor was 405 K; the temperature was not recorded for the microwave heated reactor. Reaction rate is in units of $\mu\text{mol/s/g}$.

catalysts, albeit at temperatures different from those seen in the microwave heated catalyst. From our experiments, the initial erroneous conclusion was that the CO oxidation reaction was occurring at a higher rate when heated with microwaves than with a conventional heat source (Figs. 4 and 7). However, once the temperature measurement errors in the reactor had been addressed, our final conclusion was that there is no specific microwave effect in the CO oxidation reaction over Al_2O_3 -supported Pd.

Measurement of catalyst temperature during microwave heating is a daunting task (20, 24). An optical probe would appear to be a logical choice, however in our experiments there was a problem with the optical probe we used. When an optical pyrometer was used to measure temperature from the radial direction, a potential source of error was that the observed temperature profile was averaged in the radial direction, which is the direction of heat loss to the surroundings. Therefore, the measured temperature would be an average of the reactor surface temperature and its internal temperature. The temperature measurement would be further complicated by the different emissivities of the various constituents of the heterogeneous catalyst. Due to these problems with the optical probe, we decided to use a thermocouple for temperature measurement.

The advantage of a thermocouple is that it can be inserted into the catalyst bed. While there are reports (18, 19, 24) that a thermocouple introduced normal to the direction of the microwave electric field does not influence the electromagnetic field distribution, other researchers (e.g., Chen (10)) have concluded that the interference of the electromagnetic field with the metallic thermocouples makes them unsuitable for use in a microwave heated reactor. Seyfried *et al.* (25) used a thermocouple *in situ* to measure the bulk catalyst temperature. The authors determined the error in temperature was 5–8 K while considering “limitations inherent in the experimental setup.” In addition, Roussy *et al.* (17) used a thermocouple *in situ* to measure the temperature in a fluidized bed with no apparent ill effects. However, our efforts to attain an accurate temperature measurement using a thermocouple *in situ* were unsuccessful. Therefore, in our experiments, we resorted to the use of a thermocouple that is inserted into the reactor after switching off of the microwave field. Admittedly, this is not a perfect probe since a temperature correction is necessary to account for the cooling of the catalyst bed upon contact with the thermocouple. However, after applying the correction procedure described in the Appendix, the measured temperature is within ± 5 K of the actual temperature.

Even with this method, it is important to eliminate large scale temperature gradients within the reactor. The large temperature gradients seen by the thermal imaging camera with an integral bed reactor, and the placement of the thermocouple in the bed, caused the temperature during microwave heating to be underestimated, leading to the

erroneous conclusion of a microwave enhancement in the rate of CO oxidation (Fig. 4). When a small amount of catalyst was sandwiched between two beds of inert α -alumina to form a differential reactor bed, there were still problems in obtaining an accurate temperature measurement. The time required for the isolated γ - Al_2O_3 particles to cool to the surrounding temperature would be much less than the time required to insert the thermocouple. Because of the rapid cooling, the temperature recorded by the thermocouple would be weighted towards the larger mass of the γ -alumina, underestimating the reaction temperature and yielding the apparent rate enhancement shown in Fig. 7.

These problems in temperature measurement prompted us to modify the differential bed so that the catalyst was sandwiched between a large mass of identical γ -alumina support. The comparable dielectric properties of the catalyst and the support would reduce the temperature variations and make it easier for the thermocouple to read the true catalyst temperature. With this modified differential bed reactor, our results show that the reaction rate in the microwave heated reactor is marginally greater than the rate in a conventional reactor. The reaction order in CO and the activation energy for the reaction was also unaffected by microwave heating. Since the apparent kinetics are not affected by the microwave field, it may be reasonable to assume that the CO oxidation reaction serves as an indirect probe of the metal particle temperature. On this basis, the marginally higher reaction rate in the microwave heated reactor could be caused by the catalyst being 13 K hotter than the temperature recorded by the thermocouple. There are several possible explanations for the higher reaction rate during microwave heating. The microwave heated reactor was insulated with quartz wool, and would be losing heat outward in the radial direction. The conventional reactor, on the other hand, is heated from the outside and hence the temperature variation in the radial direction would be expected to be less pronounced. This factor alone could cause the temperature recorded by the thermocouple insertion technique to be lower than the actual bed temperature. The inability of the temperature correction procedure to correctly predict the bed temperature could be another cause for the observed discrepancy. We feel both these factors could certainly explain the marginally higher reaction rates observed during microwave heating.

Other reports in the literature (6, 7, 10, 24) have also documented increased reaction rates with microwave heating. For example, Chen *et al.* (10) report that for comparable conversion, their microwave-heated reactor operated at a lower temperature than the conventional reactor. They used an infrared pyrometer for temperature measurement. As discussed above, a radial temperature profile (hotter interior and lower surface temperature) may account for the observed lower operating temperature during microwave

heating. While experimental conditions and the methods of temperature measurement differ among the various studies of microwave-assisted catalysis, we feel that difficulties in temperature measurement could certainly result in an apparent increase in reaction rates during microwave heating of heterogeneous catalysts. For example, Gourari *et al.* (24) also performed a comparative study (microwave versus conventional) of ethylene oxidation and also found a temperature discrepancy, approximately 8 K. In their case, the temperature was measured with a fine thermocouple inserted into a thin silica tube transverse to the microwave electric field, and the true temperature derived via a mathematical model. These authors suggest that the discrepancy was due to an electromagnetic effect on the kinetics of the reaction, while we feel our 13 K discrepancy is due to an indirect approach to temperature measurement and thermal gradients within the catalyst bed. It should be noted that Gourari *et al.* (24) were not able to directly measure the true surface temperature and had to rely on a mathematical model. The direct measurement of temperature in a microwave heated catalyst still represents a formidable challenge.

Of greater interest are the reported selectivity differences during microwave heating of catalysts. These selectivity differences have been attributed to a temperature gradient between the catalyst surface and the gas phase, which could modify the selectivity for reactions such as oxidative coupling of methane (10). While it is plausible that the different dielectric properties of the metal catalyst and the insulating support may cause differential microwave absorption and heating, there is some question of whether a large gradient can be sustained between the catalyst surface and the gas phase or the support. The work of Holstein and Boudart (26) shows, based on calculations, that no appreciable temperature difference is possible between a metal crystallite and the surrounding gas phase, even with the most exothermic reaction. Our results (27) agree with these calculations indicating that the metal crystallites are not significantly hotter than the surrounding support, when microwave heating is used as an energy source. While temperature gradients between the active catalyst and the gas phase or the support seem unlikely, the unique mode of energy transfer may represent one of the potential benefits of microwave catalysis, particularly for an endothermic reaction.

SUMMARY AND CONCLUSIONS

The kinetics of CO oxidation have been studied using microwave and conventional heating of an identical packed bed reactor. The temperature was measured by insertion of a thermocouple into the bed after microwave power was switched off. Optical probes were found to be unsuitable for temperature measurement under our conditions. The reactor configuration and the nonisothermal nature of the

catalyst bed influenced the accuracy of temperature measurement and two of the three reactor configurations led to the erroneous conclusion that microwave heating caused significant rate enhancements for CO oxidation. From the standpoint of accurate temperature measurement, the best configuration was a differential reactor bed containing the active catalyst sandwiched between 1-g beds of blank γ -alumina support. Our observations indicate that there was no microwave specific effect on the kinetics of CO oxidation on 5-wt% Pd/Al₂O₃. The activation energy for CO oxidation as well as the order of reaction with respect to CO was similar to that observed during conventional heating. At any given temperature, the CO oxidation rate was marginally greater with microwave heating than with a conventional clam-shell furnace. However, we feel that errors in temperature measurement could easily account for the marginal increase in reactivity observed during microwave heating. The CO oxidation reactivity serves as an *in situ* probe of metal particle temperature and suggests that the metal particles in the supported catalyst are no hotter than their surroundings during microwave heating.

APPENDIX: THERMOCOUPLE CORRECTION AND ERROR ANALYSIS

As described previously, temperature measurements were made by quenching the microwave field and immediately inserting a thermocouple into the packed bed. The temperature history was recorded on a strip chart recorder and the curve was extrapolated to the initial temperature. Fundamentally, the cooling curve represents three processes: (1) the cooling of the packed bed due to insertion of the thermocouple, (2) heating of the thermocouple until equilibrium with the bed was achieved, and (3) cooling of the thermocouple/packed bed system. Therefore, extrapolation yields a false, but consistent, temperature due to the presence of the thermocouple. To account for the deviation, a correction procedure was employed.

The conventional tube furnace was fitted so that the temperature could be measured using the fixed controller thermocouple and also with the insertion apparatus. To begin, the tube furnace was allowed to reach an equilibrium temperature as governed by the temperature controller. The power was then doused, the furnace was opened to expose the reactor tube to air, and the thermocouple was inserted. In the usual manner, the temperature history was recorded and extrapolated to obtain the initial bed temperature. In this manner, the known controller temperature could be plotted against the unknown extrapolated temperature as shown in Fig. 2. A best fit straight line through these points yields the following corrective function:

$$T_r = 1.19T_e - 15.97, \quad [1]$$

where T_r is the true temperature and T_e is the extrapolated

temperature. This expression was applied to all of the data obtained with the extrapolation technique.

An error analysis was performed by repeatedly measuring a fixed, known temperature using the procedure described above. The furnace controller was set to 260 C and the measurement was repeated 10 times. This produced a maximum range of 5 C. The total error was estimated to be ± 5 C.

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