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An examination of microwave heating to enhance diesel soot combustion

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Abstract

The effect of microwave heating on diesel soot combustion was studied through the use of a microwave heated thermogravimetric analyzer (TGA). A TGA was designed around a commercial multimode microwave oven so that carbon weight loss resulting from microwave heating could be measured. The oven door was modified, allowing infrared thermography to be used to measure soot combustion temperatures. Measurements suggest that the actual temperature of the oxidizing carbon (which is in the interior of the powder bed) is hotter than the surface temperature one measures by IR thermography. Thus, the apparent reduction in soot combustion temperature with microwave heating reported in some literature may be the result of the inability to measure the interior temperature of the soot/catalyst mass.

Keywords: Oxide catalysts; Diesel soot combustion; Microwave heating; Infrared thermography; Particulate filter; Thermogravimetric analysis

1. Introduction

There is a significant research effort being directed at lowering particulate emissions from diesel engines. Diesel particulate is relatively small (~2.5 μm or less), and hence is easily introduced to the body through the respiratory system. Because emissions from diesel engines may be linked to a number of respiratory problems, there are tightening government standards for permissible diesel exhaust emission levels. Diesel particulates typically consist of an uncombusted carbon core, adsorbed hydrocarbons from engine oil and fuel, adsorbed sulfates and water. In the United States, the Environmental Protection Agency (EPA) has implemented stricter highway heavy-duty diesel engine emission standards that will severely restrict allowed particulate emissions beginning in 2007. Among the leading technologies that are being considered to achieve lower emissions are particulate traps.

A diesel particulate filter (DPF) system employing catalysts consists of a filter material positioned in the exhaust that is designed to collect solid and liquid particulate emissions, while allowing the exhaust gases to pass through the system. Potential filter materials include ceramic foams or monoliths. Removing particulate matter from diesel exhaust is relatively simple. However, the need for filter regeneration complicates the filter technology. The volume of particulate matter generated by a diesel engine is sufficient to fill up and plug a filter over time. Hence, a means of removing the collected particulate must be provided, necessitating the development of filter regeneration technology. Regeneration can be accomplished by combusting (oxidizing) the soot to form CO_2, thereby cleaning the filter. In the absence of any combustion activation mechanism, the soot in the filter must attain a temperature in the range of 600–650 °C in order to auto-ignite and sustain the combustion. Unfortunately, the engine exhaust temperature does not typically achieve these levels, and therefore is not sufficient to initiate and sustain regeneration of the filters. A complicating factor is that the exhaust conditions can be transient, with low temperatures being the norm. Hence, a means of heating the exhaust gas or the filter bed needs to be provided. Various schemes are being considered by engine manufacturers for DPF heating including the use of microwaves [1–5].

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Microwave heating for soot filter regeneration offers the advantage of rapid heating and the possibility of more uniform filter heating than can be achieved by resistance heating. In addition, there are claims that microwave heating can result in lower soot combustion temperatures than realized by other heating techniques [6].

Microwave heating has been suggested to be beneficial in a range of applications. Several excellent reviews are available relative to sintering of oxides and metals [7–9], and heterogeneous gas phase catalysis [10,11]. In particular, a recent review by Will et al. [10] is very relevant to the present work as it describes much of the controversy regarding the beneficial effects of microwave heating on chemical reactions. In the field of catalytic reactions, there are many reports of faster reaction rates and improved yields and selectivities.

Of particular interest in this regard is the report by Ma et al. [6], who reported significantly lower soot ignition temperatures with microwave heating, being on the order of 200–300 °C for some catalysts. They conclude a non-thermal or microwave effect is responsible for the improved behavior. The primary aim of the present work was to examine if microwave heating results in reduced diesel soot combustion temperatures. In order to facilitate comparisons with conventional heating, a simple microwave thermogravimetric analyzer (TGA) designed around a commercial multimode microwave oven was built and employed in this work. In this paper, we describe the design and characterization of the microwave TGA and its use in the study of diesel soot combustion.

2. Experimental methods

2.1. Development of a microwave TGA for the study of soot combustion catalysis

A microwave TGA was developed using a commercial multimode microwave oven. The main purpose for constructing a microwave TGA was to permit direct comparison with conventional TGA measurements, which are widely used to characterize soot catalyst performance. A schematic drawing of the microwave TGA set-up is shown in Fig. 1. The design and use of microwave-based thermal analysis instruments have been reported previously [12–14]. Most recently, a series of articles by Parkes et al. describes the design and use of a sophisticated single-mode microwave-based DTA [15–18].

One of the difficulties documented in the microwave heating literature is accurate temperature measurement. Use of a thermocouple can disturb the microwave field [19]. Expensive optical fiber-based pyrometers have been developed for such temperature measurement. We chose to use infrared thermography due to its relative simplicity and ready availability of the equipment. Several reports by food industry researchers describe infrared thermography measurements as applied to microwave oven heating [20–22]. Ibarra et al. measured the temperature with an IR camera immediately after cooking [20]. This is an unsatisfactory approach for the present project. Instead we adopted an approach similar to that of Goedeken et al. [21] in that we modified the oven to allow the camera to image the oven interior.

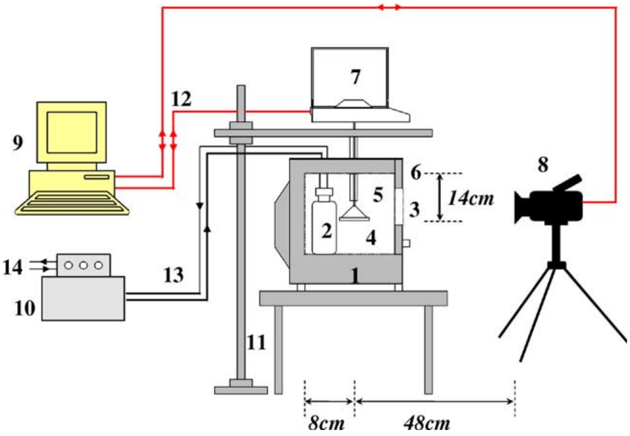


Fig. 1. Schematic of the microwave TGA: (1) sharp 2100W/R-25JT microwave, (2) cooling water reservoir, (3) CaF_2 single-crystal window, (4) quartz sample holder, (5) quartz sleeve and hanging rod, (6) modified microwave door, (7) GR-120 AD balance, (8) prism DS IR camera, (9) computer, (10) pump, (11) steel stand, (12) input and output cables connecting computer/camera and computer/balance, (13) plastic tubing for input and output water and (14) connection to house water supply.

A 2100 W dual magnetron microwave oven (Sharp Model R25-JT) was modified for these experiments In order to allow measurement of the sample temperature with an IR camera, a hole (2.75 in. diameter) was cut in both the mesh shielding and the oven window (plexiglas was substituted for the original glass). A copper screen with larger wire pitch than the original shielding screen was used to cover the hole cut in the shielding, while a CaF_2 single-crystal window was fitted into the Plexiglas. The copper screen was bonded to the original mesh with silver epoxy. The copper mesh and bond area was coated with ECCOSORB coating 300 (Emerson & Cumming) to help further decrease the microwave leakage. There is still more leakage than permitted by federal standards for a consumer oven, so personnel are moved from the immediate vicinity when experiments are performed for safety reasons. A series of experiments were performed to find the optimum mesh size. While larger mesh sizes permit easier observation, they also permit more microwave leakage. Thus, a balance was struck between ease of observation and leakage. The CaF_2 window was used to maintain the designed air-flow path in the oven.

A plastic bottle of water was placed inside the microwave cavity to serve as a load. Varying the water level alters the maximum temperature and heating rate achieved in the oven. Use of the water ballast allowed the oven to be run at full power, avoiding use of the crude power control inherent in consumer-grade ovens, while alleviating the need to integrate a more sophisticated control system such as are offered on “laboratory grade” multimode microwave ovens (e.g. CEM

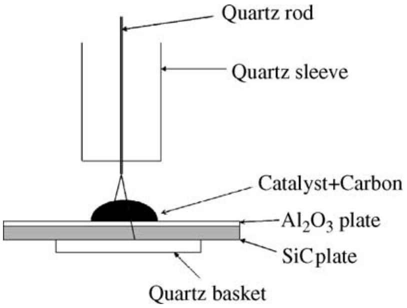


Fig. 2. Schematic of the sample holding assembly in the TGA.

Corp.). Holes drilled in the oven wall allow two sections of plastic tubing to connect the plastic bottle to a heat exchanger (chiller) to give a closed loop system. The water-flow rate and temperature can be controlled by the chiller. The use of the water load was found to decrease the microwave leakage as well as permit better control of the heating rate. It is a critical parameter in controlling the system behavior. For example, certain volumes of water necessitated use of a susceptor to heat the samples to 600 °C.

To maximize system stability, the balance rests on a steel frame, which does not contact the microwave, so there is minimal vibration of the balance during the heating process. The sample is held in a quartz basket, which is hung from a quartz rod connected to a laboratory balance (A&D GR-120). This permits sample weight to be recorded during oven operation. The quartz rod is shielded by a quartz sleeve to minimize swaying due to oven air circulation. This is shown schematically in Fig. 2. The SiC susceptor indicated in figure was only used at high water loads.

2.2. *Temperature measurement and calibration*

The temperature of the sample is measured with an IR camera (Flir Prism DS). The IR camera is used to take a picture of the sample, which is saved onto a flash memory card for subsequent analysis. Pictures are taken periodically (e.g. every 2 s) and analyzed to determine temperature as a function of time. Because the temperature is being measured through the copper mesh, there is a deviation between the measured IR temperature and the actual temperature. A correction factor for the temperature was established by comparison with the temperature measured by a thermocouple. As noted earlier, thermocouple measurements during actual microwave operation are usually troublesome due to microwave field disturbance caused by the presence of the thermocouple. To avoid this complication, a small resistively heated furnace was put into the microwave and a thermocouple was inserted into the furnace. The use of the furnace permitted a stable temperature to be established and measured both by IR observation through the screen and the thermocouple. By measuring temperatures at various furnace power levels, a temperature correction curve was determined for IR observations through the screen.

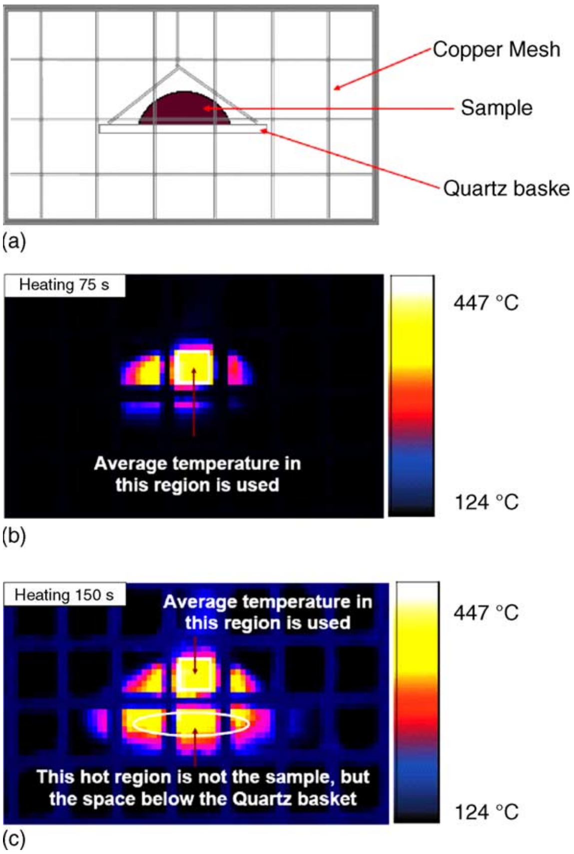


Fig. 3. (a) A sketch of the relative position of the sample, quartz sample holder and the copper mesh on the microwave door in a typical IR thermograph. Picture snapped (Pechini-750) is heated. (b) The IR image acquired of 0.1800 g carbon + $LiFe_5O_8$ after heating for 75 s, when the carbon in the mixture has begun to oxidize. The white outlined square region is chosen for analysis, and the software gives the maximum, minimum and average temperature in this region. A series of IR pictures for this experiment are analyzed based on the exactly the same region. (c) The same sample as in (b) shown after heating for 150 s. Now the heat generated by sample is being lost to the surroundings, resulting in elevated temperatures below the sample holder.

Fig. 3 shows the actual measurement configuration in more detail. In Fig. 3a, the schematic illustration shows what the camera views during a typical measurement. Fig. 3b shows the camera image acquired during a run when 0.1800 g carbon + $LiFe_5O_8$ was heated. The outline of the copper mesh is visible. Measurements were taken looking through the mesh, with the average of all points within the hottest mesh “pixel” being used to determine an average temperature. With increased heating times (Fig. 3c), the region below the sample support basket becomes hot, but these were ignored in the sample measurement. Finally, the average temperature as a function of time is what appears in the plots that follow.

3. Weight measurement

The balance (A&D GR-120) was coupled to a computer for data recording (0.2 s/point) yielding a relationship of weight and time. Because the balance in the microwave TGA is not as sensitive as a microbalance used in a conventional TGA, the carbon/catalyst ratio used is different. In conventional TGA, measurements performed in our laboratory (TA Instruments 2950), the catalyst/carbon weight ratio is ∼9:1, while in the microwave TGA, it is ∼1:1. After obtaining the temperature–time and weight–time relations, the weight–temperature relation is extracted via a spreadsheet/plotting program.

All experiments utilize synthetic soot, rather than soot captured from actual diesel engine operation. Studies have shown that a particular soot (Printex-U from Degussa) approximates diesel soot for laboratory experimental purposes [23]. This is industrial flame soot produced by high temperature pyrolysis. The nature of diesel soot can vary as a function of engine operating conditions, but Printex-U has yielded good correlation with observations on actual soot.

4. Results and discussion

Prior to studying the catalytic combustion of carbon soot, the behavior of carbon soot alone was examined in the microwave system. The first challenge was to heat the carbon soot above 600 °C at a relatively slow rate. In our initial configuration, which was highly non-optimal due to the hole in the door combined with an excess water load, carbon soot could not be heated to 600 °C, even with heating times as long as 10 min. Hence, a SiC susceptor was utilized to aid in heating. Several different susceptors were investigated, with a sample plate obtained from CEM Corp. ultimately exhibiting the best behavior. A series of trial and error experiments were used to determine the best combination of susceptor size, placement, water load position and volume and sample position. Ultimately, a set of optimal conditions was established for use in our experiments.

In a series of subsequent experiments, an oxide/carbon mixture or carbon alone was placed on a thin Al_2O_3 substrate on the SiC substrate (as shown in Fig. 2). The alumina plate was used to avoid reactions between the oxides and the SiC. In these experiments, the sample absorbs energy not only from the microwave field, but also by conduction from the substrate.

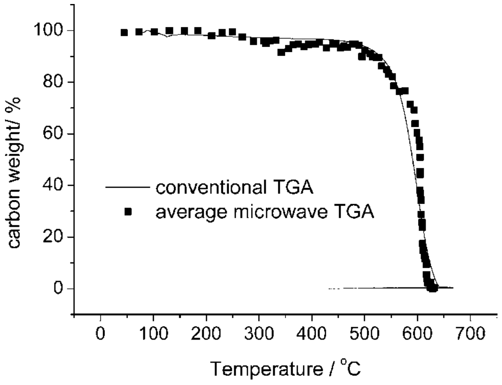
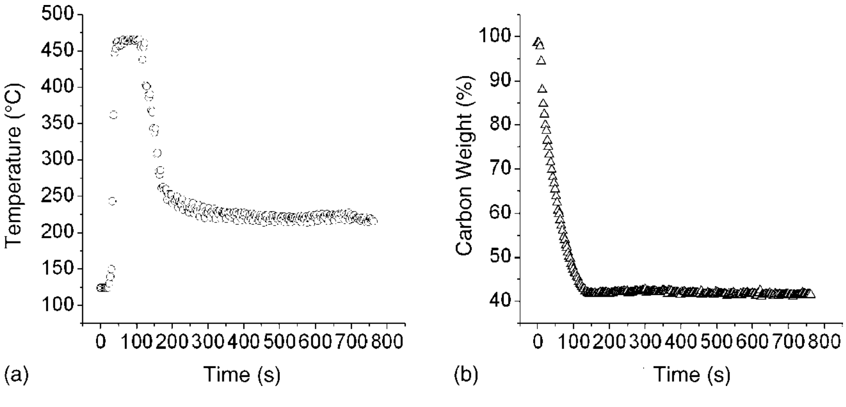


Fig. 4. The microwave TGA curve and conventional TGA curve for the combustion of carbon soot in the air.

An example of the data obtained in this configuration for heating of Degussa carbon soot on the SiC (after necessary temperature and weight corrections) is shown in Fig. 4, along with superimposed data from the conventional TGA. This curve was obtained with ∼4 min of heating in the microwave field. As shown in the curve, noticeable soot oxidation begins at about 600 °C, as is seen in our conventional TGA measurements (obtained at heating rate of 20 °C/min in 100 ml/min air-flow). The weight of material used in the microwave oven experiments is much greater than in the conventional TGA due to the reduced sensitivity of the balance. Despite this fact, there is still appreciable noise in the observed signal. Some of this is due to sparking and associated CO_2 release that causes carbon to be ejected from the sample holder. The comparison shows that with proper optimization, the microwave TGA data can be reliable, at least for the purposes of the present study aimed at examining microwave heating for soot combustion.

It should be noted that the temperature of soot oxidation will vary with the water load in the oven. If we decrease the water level to 700 ml and remove the SiC susceptor, carbon loss will start at an apparent temperature below 400 °C. Roughly 40% of the carbon weight loss is oxidized below



(a) (b)

Fig. 5. Temperature (a) and relative mass (b) as a function of heating time for carbon soot with no added catalyst.

450 °C, as is shown in Fig. 5. It needs to be emphasized that the IR camera measures the surface temperature of the carbon mass, which is a loose pile approximately 15 mm in diameter. The actual temperature in the center is not known. This is a key point in assessing the relative effectiveness of microwave heating. It can be seen in Fig. 5 that after the initial combustion event, there is essentially no additional carbon loss. Below some critical mass, thermal loss to the surroundings essentially stops any further oxidation.

When only carbon soot is heated, it is found that the observed maximum surface temperature of the carbon powder increases with the powder mass. Moreover, it is observed that the percent of the original mass that is combusted increases with total mass, since the surface-to-volume ratio decreases. Thus, for microwave heating of carbon alone in our TGA system, with increasing mass above a critical level, both the maximum observed temperature and the extent of weight loss (i.e. carbon oxidation) both increase.

The effect of the overall surface-to-volume ratio was further illustrated by dividing a pile of carbon (~0.0380 g) into two equivalent smaller piles (0.0190 g each). The result observed was that the temperature of the carbon and the amount of oxidized carbon decreased noticeably. When two piles were formed, the overall pile surface area increased, heat loss increased and heating of the mass became more difficult.

Combining the two plots from Fig. 5, yields a curve for weight loss as a function of temperature, as shown in Fig. 6 for both carbon (from Fig. 5) and a CuO + C "tight contact" mixture. "Tight contact" is the term used in the literature to describe intimate contact between soot and catalyst particles that is achieved in the laboratory by milling the soot and catalyst together [23]. It is evident from this figure that a characteristic (surface) temperature decrease follows the weight loss. The temperature decrease observed for both cases suggests that an interior core, which is hotter than the surface, experiences carbon oxidation. For the pure carbon case, after the carbon combustion in the interior is complete, the surface temperature decreases. For the CuO + C mixture, very little carbon loss precedes the beginning of the slower surface temperature decrease. The addition of CuO allows for more thorough carbon combustion, although at a slightly slower rate. The observed carbon weight loss at ~400 °C is even greater than with carbon alone (~60% of the total carbon mass is lost with CuO compared to ~40% without CuO). CuO–soot mixtures heated in the conventional TGA yield a soot decomposition temperature of ~490 °C [24]. Thus, an apparent decrease of soot combustion from 490 to 400 °C is observed with microwave heating of the CuO–C mixture. However, the 400 °C temperature measured with the IR camera is clearly not the true temperature in the interior of the heated mass.

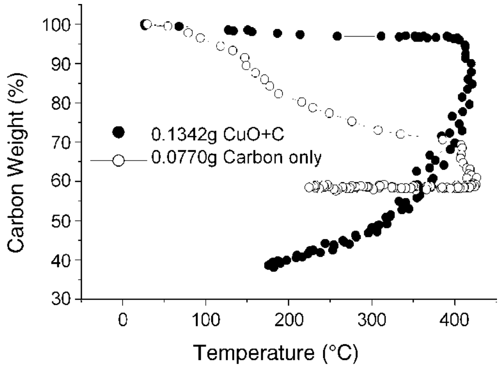


Fig. 6. Weight loss vs. temperature during microwave heating of Degussa soot and a CuO + soot mixture.

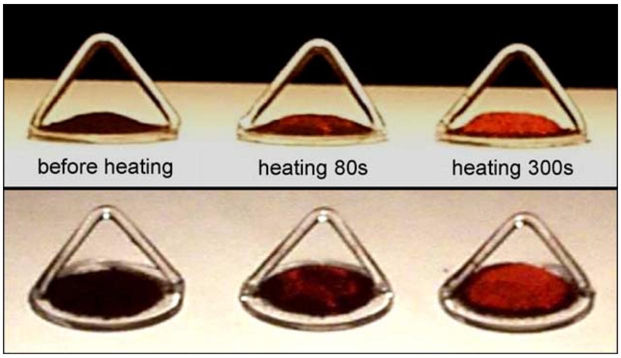


Fig. 7. The observed change of carbon + Fe_2O_3 sample color with microwave heating time.

The increasing weight loss during the temperature decrease from the maximum temperature shown in Fig. 6 for the CuO + C mixture is likely what explains the data of Ma et al. [6]. They reported a T_{90} soot combustion temperature (the temperature where 90% of weight loss has occurred) for Cu supported on TiO_2 that is 250 °C lower than the T_{50} temperature (the temperature where 50% of weight loss has occurred) for microwave heating. For resistive heating, their T_{50} and T_{90} temperatures were 420 and 510 °C, respectively. If T_{90} is less than T_{50}, there is clearly a temperature measurement problem. The T_{90} reduction from 510 to 140 °C (a change of 370 °C) reported in their work is the result of temperature measurement difficulties.

Further evidence in support of the interior heating effect was provided by studies on microwave heating of carbon + Fe_2O_3 mixtures (1:1, w/w ratio). Fig. 7 shows the samples on the quartz baskets before heating, after heating for 80 s, and after heating for 5 min. Before heating the entire sample has a reddish black color, reflecting the colors of the red Fe_2O_3 and the soot. After heating for 80 s, only a thin top layer and outer periphery of the sample are reddish black, while much of the interior is an obvious red color. The red color is indicative of the occurrence of carbon combustion, leaving only the red Fe_2O_3 behind. Finally, in the sample heated for 5 min, only the outer rim shows signs of remaining carbon. This is interpreted as clear evidence that the carbon in the interior core ignites first, and then the reaction spreads toward outside of the pile. After 5 min of heating only the thinnest regions, i.e. those most susceptible to thermal effects from the quartz, remain unreacted.

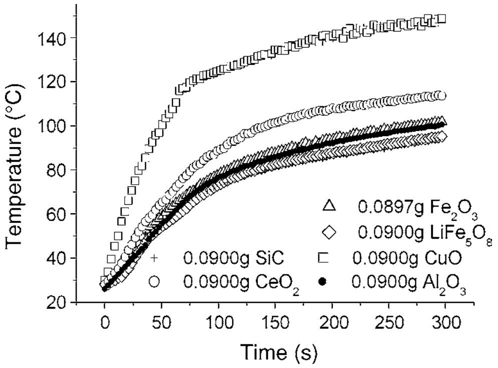


Fig. 8. Temperature change of various oxides with heating in the microwave TGA.

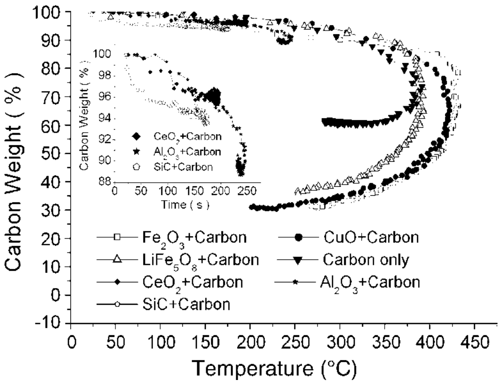


Fig. 9. Carbon weight loss as a function of temperature for various oxide–soot mixtures heated in the microwave TGA.

As noted above for carbon alone, an increase in the CuO + C heated mass leads to an increase in observed temperature. The mass dependence is common across all the systems examined, and appears to be an artifact of the decreasing surface-to-volume ratio. The carbon mass loss increases as the total CuO + C mass increases, due to the corresponding decrease in relative surface area.

Additional systems were characterized for their microwave heating behavior, including Ce_2O_3 + C, Al_2O_3 + C, $LiFe_5O_8$ + C and SiC + C. Fig. 8 shows the temperature change observed during microwave heating of the oxides used in these studies in the absence of carbon. CuO showed the best coupling to the microwave field. When Fe_2O_3 or SiC (400 mesh) were heated (cooling water volume is 700 ml), they exhibited similar heating rates, as judged by the surface temperature.

The relative performance of all of the compounds considered in promoting soot combustion in the microwave TGA is shown in Fig. 9. In all cases, the amount of carbon was the same and the carbon/oxide weight ratio was 1:1. The inset in this figure shows the results for the relatively non-reactive SiC, Al_2O_3 and CeO_2 compounds. As seen in figure, CuO– and Fe_2O_3–soot mixtures achieve the highest temperatures (∼425 °C) followed by $LiFe_5O_8$ + C and soot alone (∼375 °C). Much lower temperatures (and practically no carbon loss) were attained by the CeO_2–, SiC– and Al_2O_3–soot mixtures. SiC is a widely used microwave susceptor, but it does not act as a carbon oxidation catalyst, while Al_2O_3 is neither a microwave susceptor nor a catalyst. None of these three compounds resulted in more than ∼10% carbon loss.

Both Fe_2O_3 and SiC behaved similarly when heated in the microwave field in the absence of carbon. However, when they were mixed with carbon, the Fe_2O_3 showed much greater carbon combustion than SiC, due to the catalytic effect of the Fe_2O_3. The SiC + C mixture showed very little soot oxidation, with the temperature remaining below 200°, while the Fe_2O_3–soot mixture achieved much higher temperatures and resulted in a 70% carbon loss. This illustrates that effective catalysts contribute to a reduction in oxidation temperature in the microwave TGA, similar to the effects seen in conventional TGA heating. The fact that carbon alone easily combusts, whereas carbon in contact with SiC powder does not indicates the complex nature of the interaction between the soot particles and the oxide/carbide powder particles. Clearly some energy "heat-sinking" by the added powder particles occurs, resulting in poor soot combustion relative to the carbon-only case. The higher temperatures achieved by CuO– and Fe_2O_3–soot mixtures relative to the carbon-only sample is likely due to a combination of powder particle heating through coupling with the microwave field along with enhanced soot oxidization.

The three best compounds among those considered were the CuO, Fe_2O_3 and the spinel $LiFe_5O_8$, with all three of the compounds resulting in relatively similar carbon weight losses. The combustion of soot alone occurs at a lower

Table 1
Conventional and microwave TGA results for various oxide/carbon mixtures in flowing air

Mixture (1:1 oxide/carbon weight ratio)	Conventional TGA		Microwave TGA	
	T_{start} (°C)	T_{max} (°C)	T_{start} (°C)[a]	T_{max} (°C)[b]
Al_2O_3 + C	545	577	245	250
SiC + C	535	554		175
CuO + C	491	495	330	450
Fe_2O_3 + C	501	531	270	470
CeO_2 + C	550	576		200
$LiFe_5O_8$ + C	450	501	345	380
Carbon soot	553	599	295	380

[a] Apparent T_{start}, the temperature at which there is a 10% loss of carbon.

[b] Apparent T_{max}, the maximum temperature observed during combustion.

apparent surface temperature than in the presence of these compounds, yet all three of these compounds lead to more complete combustion of the carbon.

The carbon soot combustion characteristics of the compounds in the microwave system relative to that seen in the conventional TGA are summarized in Table 1. As emphasized earlier, comparing conventional TGA results with the microwave results from this study is actually a comparison of the surface temperature in the microwave TGA with what better approximates a bulk temperature in the conventional TGA. For example, although both $LiFe_5O_8$ and CuO appear to yield lower soot decomposition temperatures in the presence of microwave heating, this is a fallacious comparison in the present experiments because of the different temperature measuring techniques employed. Attempts in this study to heat only a thin layer of powder (so there is no “interior”) were unsuccessful. Thin layers cannot be heated to elevated temperatures without the use of a susceptor due to excessive heat loss to the surroundings (support basket and air). Moreover, we lack the ability to measure local temperatures at contact points between soot and oxide particles. Even if one were able to measure interior temperatures, it is quite likely that temperatures would not be uniform due to the formation of “hot spots”. Hot spots can form due to variability in particle contact, microwave field non-uniformities, etc. Gross evidence of hot spots in some cases was provided by sparking activity. Hot spot formation, coupled with problematic temperature determination is likely the primary source for the confusion about microwave effects [10,25,26]. In the present case, we believe the apparent decrease in soot combustion temperature with microwave heating is an artifact of the inability to measure local temperatures in the combusting powder mass.

The formation of hot spots as described in the present work is an impediment for the application of a microwave TGA to examine particulate materials. Although microwaves are attractive as a means of heating samples from the inside out [12], the difficulty in assuring uniform heating and achieving accurate temperature measurements pose great challenges. The best approach to applying a microwave TGA to a wide range of materials and minimize hot spot effects are likely by having the sample rest on a susceptor. This will allow for more uniform heating and aid in temperature measurement. A microwave TGA would be most advantageous as a means to study microwave enhanced reaction kinetics, which is a subject of continuing interest.

5. Conclusions

A TGA system designed around a conventional multimode oven was successfully implemented and used for the study of diesel soot combustion. These studies show that claims of reduced soot oxidation temperatures in resulting from microwave heating may be the result of the difficulty of measuring actual local temperatures in the catalyst soot powder bed. Measurements in this study indicate that the actual temperature of the oxidizing carbon (located in the interior of the powder bed) is hotter than the surface temperature measured by IR thermography. Thus, the apparent reduction in soot combustion temperature with microwave heating may be the result of the inability to measure the interior temperature of the soot/catalyst mass.

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