FORMATION AND OXIDATION OF SOOT PARTICULATES IN A DIESEL FUEL SPRAY FLAME

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This paper presents an analysis of experimental data acquired inside the combustion chamber of a high-pressure steady-flow spray burner. The data consists of dimensions of primary particles in soot agglomerate samples taken from axial locations at different distances from the injector tip and at off-axis positions at a fixed axial distance mid-way along the chamber. Corresponding values of soot volume fraction are given. Diesel fuel was used in these tests, which were conducted at pressures between 1.1 and 2.1 MPa with input mixture equivalence ratios ranging from 0.8 to 1.2.

Particle size distribution and mean particle diameters were calculated at the different locations using transmitting electron microscopy and associated image-analysis diagnostics. A computer-simulated oxidation process was applied to measured size distributions from upstream locations to compare with actual measured distributions obtained at downstream locations. Agreement was found to depend on local conditions.

Evidence of internal oxidative attack is demonstrated for some individual primary particles within the agglomerates through a widening of the crystallite spacing under certain operating conditions.

\textit{Keywords:} Soot; Particulates, Oxidation kinetics; Size distribution

INTRODUCTION

The importance of minimizing the emissions of combustion-generated pollutants and their adverse effects on the environment presents researchers with the task of finding means to improve combustion processes. For diesel engines, this specifically relates to the perceived problem of smoke emissions. A more widespread acceptance for urban private transportation has been limited by public concern over the emission of dark exhaust smoke and its possible carcinogenic properties. This disadvantage has overshadowed the advantages of its fuel tolerance, efficiency, and lower gaseous pollutant emissions.

The combustion processes in the diesel engine are highly heterogeneous in nature and involve the introduction of an atomized liquid fuel spray into an oxidizing environment, with changing properties, in a finite time. The ability to pre-
dict the spatial and temporal variations of flow properties and chemical species in such a system is of vital importance to its evolution. In developing computer models, the need for validation is important and, therefore, there is a need for precise data. Prediction of diesel engine exhaust-smoke levels requires a knowledge of the formation and oxidation mechanisms of carbon particulates and an understanding of the effect of the governing properties on the rates of these processes.

Measurements of particulate matter inside the combustion chamber of a running diesel engine are difficult to make and complex to interpret because of the problems of accessibility, limited time, and spatial and cyclic variations of the properties. Exhaust-gas measurements, while easier to make, are difficult to correlate with in-cylinder events and conditions. Premixed and non-premixed laboratory gas flames offer precisely controlled environments in which to measure properties, but conditions are usually remote from those prevailing in diesel engine operation. Combustion-bomb, shock-tube, and rapid-compression-machine experiments provide single events at more appropriate conditions, but have much the same spatial and time limitations as engine tests. Soot volume fraction may also be sufficiently high for light obstruction to be a problem with optical measurement techniques at elevated pressures. In such cases, tried-and-tested methods must be resorted to, such as gas withdrawal with sampling probes or thermophoretic sampling.

For a number of years, experimental work has been conducted on spray combustion at elevated pressures in the Department of Engineering at QMUL. To avoid the limitations imposed by the geometry and cyclic changes of properties in diesel combustion, a steady-flow combustor has been designed to operate at pressures of up to 10 MPa. This work is linked to building an understanding of the processes, kinetic mechanisms, and governing parameters in the formation and removal of carbonaceous particulate matter in high-pressure heterogeneous liquid hydrocarbon combustion. The rationale of this approach is to provide most of the engine operating conditions: spray atomization, liquid evaporation, gaseous mixing, spontaneous ignition, combustion, and soot generation in different environments at elevated pressures but with an extended time-scale to permit analysis. It is relevant, but not exclusively limited, to diesel engine spray combustion.

**EXPERIMENTAL TECHNIQUE**

The system enables gaseous and particulate samples to be taken from the exhaust of the combustor or from locations along the axis and radially displaced from the axis inside the spray flame. The fuel and airflow rates may be controlled and the exhaust area varied to enable a range of steady-state conditions of different input equivalence ratio, and pressure to be maintained. The high-pressure combustion
facility features an interlocking modular water-cooled, stainless-steel, confined spray-burner assembly with associated air and fuel supply and control systems. There are twin particle traps and gaseous-emission-measuring instruments in the sample line. By close control of the fuel and air supply properties and the chamber exhaust outlet area (by means of a nozzle-plunger arrangement), the overall input mixture ratio and chamber pressure are set and held steady. The fluctuation-damped air supply is from a high-pressure receiver, charged to 6.5 MPa by a three-stage compressor with associated intercooler, moisture-settling chamber, and dryer. The fuel is supplied from a multi-cylinder fuel pump with damped, common-rail output at 20 MPa through a single-hole (0.2 mm diameter), continuous-spray, pressure-jet injector. This is aligned along the chamber axis (with a 10° cone angle) in the co-flowing uniform airstream. In all the experimental work described here, the fuel used was Shell diesel fuel, having a density of 830 kg/m³ and C:H ratio 6.5:1 by mass and an effective mean molecular formula $C_nH_{1.86n}$ where $n$ is the number of C atoms. Gaseous and particulate emissions were sampled from the exhaust section through the nozzle-plunger device and from inside the spray flame through the chamber wall of an axially relocatable chamber unit. Water-cooled stainless-steel sample probes were used for this purpose. Depending on the chosen operating conditions, the inlet air temperature being ambient, maximum chamber temperatures ranged from 1700 to 1900 K, with exhaust temperatures about 700 to 800 K.

Steady operating conditions were first established (and continuously recorded) by observation of the high-frequency data-logged gaseous combustion-product composition and temperature. Particulate samples were then collected in filter units from the metered gaseous sample in recorded time intervals (of the order of two minutes) prior to making precise mass determination using a sensitive balance. Repeat determinations were made in separate runs, in which essentially the same operating conditions were established. The system has been further described elsewhere (Crookes, 1999, 2000, and 2003).

The analysis of a number of particulate samples taken from different locations along the axis and across the spray flame is presented at gauge pressures from 1 to 2 MPa and at conditions ranging from lean to rich ($\phi = 0.8$ to 1.2). For this, the samples were transferred on 3-mm diameter, 300-mesh, carbon-coated copper grids to a JEOL (JSM 2010) transmitting electron microscope fitted with a CCD camera linked to a PC equipped with image-analysis software. Electron-micrograph images with up to 500,000 times magnification were obtained. Soot particle-size analysis was performed on multi-agglomerate samples comprising several hundred primary particles.
EXPERIMENTAL RESULTS

Soot sampled from most locations within the combustion chamber was typically in the form of approximately spherical primary particles, fused together in aggregates. Carbon deposition had bonded the structure and overlayered the individual particles, masking to a greater or lesser extent the identity of the original particles. This led to a wide range of agglomerate clusters with differing numbers of particles, configurations, and particle-size distributions. Throughout the soot formation process, when conditions were suitable, oxidation also influenced the size of the particles in the agglomerates. TEM images revealed a less closely packed lattice-spacing under certain conditions than the graphitic crystallite structure normally associated with diesel soot and carbon black. A looser, but quite regular, lattice internal structure can be seen in some particles in the agglomerates shown in Figure 1a when compared with that in the primary and fused particles shown in Figures 1b and 1c. A difference in spacing of almost a factor of 2 (from about 0.34 nm to 0.62 nm) has been reported (Crookes, 2003), as seen in the middle region of the agglomerate. In some cases, the particle interior appears to be totally void (capsular), as at the upper left of the agglomerate, and possibly totally collapsed. The operating conditions under which such different morphologies were

FIGURE 1 (a) Soot agglomerate, (b) primary, and (c) fused particles sampled from inside combustion chamber.
found are, in the case of the agglomerate in Figure 1a, and the particles in Figures 1b and 1c, respectively, lean and rich.

Figure 2 illustrates the magnitude and range of primary particle diameters in samples collected at different axial locations along the chamber (which is also shown for clarity, with axial distances being from left to right) and at different radial distances from the axis at 0.41 m from the injector tip. The data is for operating conditions of 1.6 MPa and a stoichiometric overall input ratio. In most cases, the number frequency is highest between 10 and 40 nm diameter, but some particles in the size range over 100 nm were detected at off-axis locations, towards the chamber walls and chamber exit. The mass frequency, as would be expected, resulting from a cubed power of diameter relation, is highest at larger diameters than the number frequency. Values of corresponding number-mean ($D_n$) and mass-mean ($D_m$) diameters, computed from the TEM images, are tabulated for different input equivalence ratios, $\Phi_{in}$ in Table 1 at 1.6 MPa.

In the present high-pressure system, the spray flame has a symmetrical geometry spreading down the chamber around the chamber axis. The local vapor-phase equivalence ratio is rich early in the spray core as droplets evaporate, and it decays to the overall input value down and across the chamber as air is entrained, as in a diesel engine spray plume. Soot levels peak and then decay along the axis as oxidation prevails. The mean values of primary particle diameter,
under the particular conditions of Figure 2, rise slightly on balance, along the chamber and away from the axis. The size-range variation, as represented by the standard deviation ($\sigma$), also, in general, follows in the same proportion. Corresponding experimentally derived soot volume fraction, $f_v$, values, which increase in overall magnitude with input equivalence ratio, and the resultant particle number densities, $N$, are also given in Table 1. This shows that oxidation is well advanced for the stoichiometric input condition at the axial location (0.41 m) where the radial measurements were made, is more so at the lean input condition, and not so well advanced at the rich input condition. Correspondingly, the mean particle diameter rises more in the lean case and drops in the rich case, although

<table>
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<th>Distance, m</th>
<th>$f_v$, $10^{-6}$</th>
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<th>$\sigma$, nm</th>
<th>$D_m$, nm</th>
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magnitudes stay higher at the rich condition. Number-mean diameter values at 2.1 MPa, together with corresponding soot volume fractions for lean, stoichiometric, and rich cases, are shown in Figure 3, at different locations. Soot volume fraction is computed from measured soot concentration at local conditions assuming a value of $2 \times 10^3$ kg/m$^3$ for the primary soot particle density (agglomerate densities would be lower). [Repeatability of soot mass determination was in general good with greatest variation in the vicinity of rapid property gradients, for example, local gas-phase equivalence ratio, at the peak soot levels.] Measured values at the higher pressure exhibit similar trends to those described for the lower pressure. Off-axis in an annulus at 20 mm radius, there is a maximum soot volume fraction, which increases in magnitude from lean to rich conditions. Mean diameter is higher at the higher pressure as is the off-axis peak value of soot volume fraction. Spray modeling analysis by the authors and co-workers (Nazha, Ra-
(jakaruna, and Crookes, 2000) show, in iso-property maps (Crookes, 2003), that this region is one of near-stoichiometric mixture and peak temperature.

**SIMULATED OXIDATION**

During the combustion of a liquid hydrocarbon fuel, the physical processes of atomization, droplet heating and evaporation, and the subsequent mixing of the vapor with entrained air lead to an intensely nonhomogenous situation. Changing global properties like combustion-chamber pressure and input mixture ratio enhance this local variation. Within this environment, the mechanisms of soot nucleation and growth are accompanied, where conditions permit, by the process of oxidation. These conflicting mechanisms are sensitive to the same local physical and chemical properties, and designing suitable means of experimentally decoupling them is a challenge.

A number of spray combustion models (Nazha et al., 2000; Nishida and Hiroyasu, 1989), soot formation models (Narasimhan, 1964; Moss, Stewart, and Young, 1995), and oxidation models (Appleton, 1973; Lee, Thring, and Beer, 1962) are available. There is not a vast amount of relevant experimental data at elevated pressures with which to validate them. The local data provided here may be analyzed to determine if there is consistency, in relation to such models, between samples obtained from earlier positions and at subsequent locations for different operating conditions. In part, to do this, a comparison is made between measured data and measured data from upstream, used as input values for subsequent simulated oxidation using a suitable kinetic model. In this case, the widely adopted model of Lee et al., (1962) has been chosen without yet incorporating the modification of Ladommatos, Song, and Zhao (2002).

Figure 4a shows a typical particle-number size distribution from a sample taken from the axial location 0.29 m from the injector tip at a chamber pressure of 1.1 MPa and a stoichiometric input equivalence ratio. Alongside are the resultant distributions calculated from computer-simulated oxidation of the particles after about 90 and 250 ms duration, respectively. These values were taken to represent the time taken for the agglomerate inside the chamber to move to the next two axial-sampling locations, using the model of Nazha et al. (2000) in Crookes (2000). It can be seen that the effect of this simulated oxidation is to increase the proportion of smaller particles and reduce that of the larger ones while the total number decreases.

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1"Simulated oxidation" is a term used here for computer-simulated primary particle size reduction by means of a surface oxidation mechanism.
COMPARISON WITH EXPERIMENT

In Figures 4b and 4c, these computed distributions are compared with experimental values obtained under the same operating conditions as in Figure 4a, at axial locations of 0.41 m and 0.635 m, respectively, from the injector tip. While there is fair agreement at the nearer location, a correlation is not found at the further distance (at the end of the chamber) under these conditions. Here, the computed values have a much higher proportion of smaller diameter particles and fewer with larger diameters, implying that the computer-oxidation gives an overestimate.

Comparisons of measured and computer-oxidized particle size distributions were made at 1.6 MPa for lean, stoichiometric, and rich conditions. In Figure 5a, data in agglomerates sampled from the axis at 0.41 m from the injector tip are compared with computer-oxidized distributions from axial samples at 0.29 m. Reasonable agreement was found at the stoichiometric condition, as shown, and also at the rich conditions at this pressure, while an over-prediction of small particles at the lean condition was detected. Further down the chamber, Figure 5b shows a simulated oxidation of a combined distribution derived from samples taken at all sampled radial locations across the chamber at the axial distance of 0.41 m from the injector tip. In comparison with the axial-measured data at 0.635 m, there was high over-prediction of the proportion of small particles at the lean condition, closer agreement at the stoichiometric condition, and very good agreement, as is shown, at the rich condition.

In Figure 5c, the same combined distribution from 0.41 m is again compared, this time without any simulated oxidation, with the data from 0.635 m as in Figure 5b. In this case, excellent agreement is shown at the lean condition. Agreement was also close for the stoichiometric condition, but not for the rich case, in which there were more small particles in the measured local data at the further position from the injector tip than in the upstream distribution, indicating further oxidation to have occurred.

DISCUSSION

As can be clearly seen in all cases in Figure 2, there is a wide range of primary particle sizes in agglomerates sampled at different locations in the chamber. These are also within the usual size ranges quoted for flame studies (Hanisch, 1994) and diesel engine tests (Houben, Pischinger, and Lepperhoff, 1994). What is possibly surprising is that the mean values in Table 1 vary little and that they do not uniformly decrease along or across the chamber. Indeed, under some conditions, they decrease and in others they increase.
FIGURE 4  (a) Measured particle size distribution at axial distance 0.29 m from injector together with same distribution, computer oxidized to 0.41 and 0.635 m positions, at 1.1 MPa; $\phi = 1.0$. (b), (c) Same particle size distributions as in (a) compared with measured samples.
FIGURE 5 (a) Measured particle size distributions at 0.41 m at 1.6 MPa and $\phi = 1.0$ compared with computer-oxidized samples from 0.29 m at same conditions; (b) Measured particle size distributions at 0.635 m at 1.6 MPa and $\phi = 1.2$ compared with computer-oxidized samples from 0.41 m (averaged across chamber) at same conditions; (c) Measured particle size distributions at 0.635 m at 1.6 MPa and $\phi = 0.8$ compared with unoxidized distribution from 0.41 m (averaged across chamber) at same conditions.
Data from a swirl-stabilized spray combustor at atmospheric pressure (Fang, 1998) identified regions of nucleation, growth, and oxidation and suggested that cluster-to-cluster aggregation rather than surface growth was the dominant growth mechanism for aggregates. Similarly, final growth of soot volume fraction at 1.5 MPa in an ethylene flame (Hanisch, 1994) was deduced to be mainly by increased nucleation after early particle growth. While here, primary particle diameters are larger, a similar pattern in mean particle diameter variation with axial distance to that of Fang (1998) is visible in the data obtained under rich input conditions and higher pressure, in the present work. The unlikely stability or increase in number-mean particle diameter, when oxidation should be the prevailing mechanism, under lean and stoichiometric conditions, needs some explanation if it is a real or significant phenomenon. One possibility is that oxidation is completed by this stage under these conditions, and another is that the number of small particles completely disappearing exceeds the number reducing in diameter, so the denominator used to determine the mean is smaller. The comparisons shown in Figure 5 go partly towards providing an explanation of why the measured number mean particle diameters do not vary significantly in extracted samples along the spray flame axis.

If, as is sometimes assumed in combustion models, the particles were of a monosized distribution, then the mean value would gradually diminish by oxidation. This is illustrated by the line of diamonds in Figure 6, for a mono-sized sample with the same mean diameter value as measured at the axial location 0.29 m from the injector tip. This has been computer-oxidized to the end of the cham-

**FIGURE 6** Comparison of mean diameter of computer-oxidized samples from 0.29 and 0.41 m with experimental mean values at 0.41 and 0.635 m.
ber (0.635 m) at 1.6 MPa and stoichiometric input equivalence ratio. The end points of a similar exercise for the mean value of the combined sample at 0.41 m are also shown in Figure 6 as triangles. The unoxidized endpoint of the same sample (equal to the starting value) is given by a cross, and the actual measured axial values at these locations are included as squares. It is evident from Figure 6 that in this high-pressure combustor, under these conditions, oxidation has, more or less, stopped at the location of the samples taken at 0.41 m from the injector. This is apparent in the soot volume fraction measurements in Figure 3. From the data in Figures 3 and 5c the oxidation is, as might be expected, completed even earlier at the lean condition. However, in Figures 3 and 5b, it appears that oxidation is still proceeding at this stage for the richer input condition. Further work using different oxidation models has now been undertaken, together with a sensitivity analysis of the comparisons to selected property values.

Another consideration, not usually found in combustion modeling, is that of the morphological changes involved, when carbon is consumed in the oxidation process. In carbon black oxidation, observations have been made of widening intercrystallite spacing (Heckman and Harling, 1965), which were associated with penetrative oxidation. This has not been found in diesel soot oxidation in which "shelling" off of surface layers is suggested (Ishiguro, 1991) to be the normal process. A recent review by Stanmore, Brilhac, and Gilot (2001) considers penetrative oxidation to be less likely under rich conditions and higher temperatures when surface reaction, respectively by OH and O₂, would be more favored. In the present work, widening intercrystallite spacing, as seen in Figure 1a, has been observed under mainly lean conditions. However, temperatures are still higher under these conditions (Crookes, 1999) than would be expected for this kind of penetrative oxidation (Stanmore et al., 2001), and surface reaction would be expected, although it is possible that different limits apply at elevated pressure. Clearly, there is a need for further investigation with samples obtained both under controlled conditions at elevated pressure as reported here and those from within a diesel engine combustion chamber environment.

CONCLUSIONS

Results of experimental analysis of soot samples taken from inside the combustion chamber of a high pressure steady-flow spray burner presented here at pressures up to 2.1 MPa lead to the following observations:

1. Soot volume fraction displayed an axial peak value where soot formation mechanisms were subsequently overtaken and dominated by oxidation reactions. By matching particle size distributions from given locations with
upstream values subject to computer-simulated oxidation, an estimate of the effect of the prevailing state was made.

2. Under the conditions of the tests presented, oxidation was assessed to be virtually completed at the mid-chamber position for the stoichiometric input condition. Earlier and much later completion was evident under lean and rich input conditions, respectively.

3. Off-axis, in an annulus at 20 mm radius, there was a maximum soot volume fraction that increased in magnitude from lean to rich conditions and with increasing pressure. This was the region where the local mixture ratio was predicted to promote the peak temperature.

4. The primary particle diameter number frequency was found to be, in most cases, highest between 10 and 40 nm. Some particles over 100 nm were detected away from the chamber axis and, probably by mixing from cooled upstream regions, later in the flame. Number-mean primary particle diameter generally increased with input equivalence ratio and pressure.

5. Under mainly lean conditions, TEM images revealed a less closely packed lattice spacing inside some particles than the graphitic crystallite structure normally associated with diesel soot and carbon black. This wider spacing was believed to result from internal oxidation.

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References


