Visualization of soot formation from evaporating fuel films by laser-induced fluorescence and incandescence

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Abstract

Late-evaporating liquid fuel wall films are considered a major source of soot in gasoline direct-injection engines. In this study, a direct-injection model experiment was developed to visualize soot formation in the vicinity of evaporating fuel films. Isooctane is injected by a multi-hole injector into a wind tunnel with an optically accessible test section. Air flows continuously at low speed and ambient pressure through the test section. Some of the liquid fuel impinges on the quartz-glass windows and forms fuel films. After spark ignition, a turbulent flame front propagates through the chamber and ignites pool fires near the fuel films, leading to locally sooting combustion. A laser light sheet with a wavelength 532 nm excites laser-induced fluorescence (LIF) of large polycyclic aromatic hydrocarbons (PAH) with five or more aromatic rings, considered as soot precursors, near the evaporating fuel films. Additionally, a light sheet at 1064 nm excites laser-induced incandescence (LII) of soot particles. Two intensified CCD cameras simultaneously detect the LII and LIF signals, and thus visualize PAH and soot. In complementary line-of-sight imaging, the fuel spray, chemiluminescence of the flame, and soot incandescence are captured with a high-speed color camera. In addition to this fuel-injection experiment, a sooting laminar coflow flame burning ethylene in air (Santoro burner) is used for preliminary in-situ measurements. In the latter, PAH LIF is detected in a hollow cone region, indicating the transition from fuel to small PAH in the dark region and the growth of the latter into large PAH in the cone region. The edges of the PAH LIF cone are covered by regions of high soot LII signal, indicating the nucleation region where soot forms from PAH. In the wind tunnel, layers of PAH are found in the close vicinity of the evaporating fuel films. Soot forms mostly spatially separated from the PAH and with high spatial intermittency. The chemiluminescence in the bottom of the images indicates the oxidation of soot and PAH, being transported downstream.

Introduction

The formation of soot, as a solid-phase species, from polycyclic aromatic hydrocarbons (PAH), as a gas-phase species, is still barely understood. In gasoline direct-injection (GDI), evaporating fuel films and the resulting inhomogeneities of the fuel/air-mixture make the formation of polycyclic aromatic hydrocarbons and soot likely. In aliphatic flames the fuel pyrolytically decomposes into polyacetylenes, and then recombines to aromatic species by cyclisation reactions. The latter grow further due to the collision with acetylene species (HACA-mechanism) into large molecules, consisting of at least two benzene rings, known as polycyclic aromatic hydrocarbons [1-3]. The further growth of PAH leads eventually to the formation of soot particles. Optical measurement techniques for soot diagnostics are light extinction, elastic light scattering (ELS), and laser-induced incandescence (LII) [4]. In LII, a high energy laser-pulse heats soot to temperatures high above flame temperatures, leading to an intense broadband emission according to Planck’s law. LII signal is detected point-wise or 2D and is a measure of relative soot concentrations or soot volume fractions after calibration. Bockhorn investigated the formation and oxidation of soot in detail [5]. Characterizing soot and measuring particle sizes or volume fractions by means of laser-induced incandescence (LII) was subject to laminar premixed [6-8] and diffusion flames [9-11]. Geigle et al. two-dimensionally imaged the soot volume fraction in a gas-turbine model combustor and found soot to be formed with high temporal and spatial intermittency [12].

So far, the investigation of soot formation in internal combustion engines focused mainly on Diesel engines. The diffusion controlled combustion is likely for the formation of undesired soot
particles from local inhomogeneities in the fuel/air-mixture. Dec et al. investigated the soot formation in Diesel engines with 2-D imaging of soot LII [13, 14]. Dec came up with a conceptual model of the different combustion stages [15]. Skeen et al. gave a broad overview on the experimental and modeling site of soot formation under the ECN “Spray A” configuration [16]. The formation of soot from diffusion flames in a gasoline direct-injection engine, in particular from pool fires, is barely investigated. Two-dimensional soot volume fraction measurements [17] and particle size measurements [18] were carried out in GDI engines by means of LII. Francqueville et al. measured soot volume fractions in a spray-guided direct-injection spark-ignition (DISI) engine and observed a sharp decrease of the soot concentration during the expansion stroke, most likely due to oxidation.

Many PAH exhibit broadband optical absorption and fluorescence properties, which makes them, in particular in turbulent environments, favorable for non-intrusive in-situ measurements [19]. When PAH are increasing in number of benzene rings, their absorption spectrum shifts to longer wavelengths, so as the subsequent fluorescence. This allows for a size-dependent detection of PAH based on the excitation wavelength [4]. Exemplarily at flame temperature, PAH containing more than five aromatic rings absorb and fluoresce in the visible while smaller PAH also absorb and fluoresce in the UV [4]. With 266 nm most of the PAH can be electronically excited [20]. Petarca et al. found laser-induced fluorescence (LIF) spectra of PAH in an n-heptane diffusion flame becoming less structured and more red shifted with increasing HAB for a certain excitation wavelength. This indicates that a multitude of large aromatic species is excited with increasing HAB [21]. Bijaoui et al. found a red-shift of 50 nm in the fluorescence spectra collected from a diffusion flame with increasing HAB when exciting in the UV. In contrast to that, no spectral shift was found in a premixed flame when exciting in the UV. This indicates the stratification of PAH in the diffusion flame with increasing HAB and that many different size classes of PAH are excited in the UV. Extending the excitation wavelength from 415 nm to 680 nm shifts the full width half maximum (FWHM) about 100 nm to the red. Excitation in the visible did not show any shift to longer wavelengths with increasing HAB, indicating a limited pool of PAH being excited in the visible (PAH with more than five aromatic rings [4]) [22]. The high number of different PAH, their different spectroscopic properties and the dependency of those on temperature and the collision quenching environment make a quantification of PAH concentration based on LIF impossible [4, 23]. A species selective measurement of PAH is possible with ex-situ techniques like photoionization coupled with mass spectrometry or jet-cooled laser-induced fluorescence, [4, 22, 23]. Beretta et al. made an attempt to identify individual PAH based on different features in the fluorescence spectra when varying the excitation wavelength from 250 to 514.5 nm [24]. Schoemaecker et al. investigated soot formation in a diffusion flame by simultaneously visualizing PAH and soot, excited at 532 and 1064 nm, respectively [25]. Vander Wal et al. visualized both PAH LIF and LII of soot and indicated a “dark zone” between PAH and soot in a laminar diffusion flame [26]. The growth of PAH in an optically accessible diesel engine was visualized by increasing the excitation wavelength from 266 to 532 and 633 nm by Musculus et al. [27]. Simultaneously the laser-induced incandescence, excited at 1064 nm, reveals the soot inception regions. Combining PAH LIF and soot LII with the imaging of natural flame luminosity was barely done. Smyth et al. have imaged broadband fluorescence and visible flame luminosity in hydrocarbon diffusion flames to identify soot oxidation regions [28]. In the following, the denomination PAH LIF will be used, comprising the signals of large PAH, 5-membered ring PAH, such as fluoranthene and acenaphthylene, and incipient particles since all these species are known to absorb and fluoresce in the visible under flame temperatures [20, 21, 24, 29]. The purpose of this work is to present a measurement technique to simultaneously visualize PAH, excited at 532 nm, and soot, excited at 1064 nm, from combusting fuel films in a direct-injection model experiment. The sooting laminar coflow flame of a Santoro burner was employed for preliminary measurements.

Experiment

Figure 1 shows a sketch of the direct-injection model experiment, the optically accessible test section of a wind tunnel. The test section consists of three quartz walls that provide optical access. Spark electrodes and an injector protrude through an aluminum plate, the fourth wall of the section. Heated air flows continuously from the top to bottom with a nominal velocity of 1.8 m/s. To provide small-scale turbulence and a uniform bulk flow, a perforated plate stacked with fine wire meshing is located between the air heater and the test section. A six-hole nozzle injects fuel evaporating into the hot air
flow. Some of the injected fuel wets the quartz wall on the opposite side. Fuel films 2, 3, 5, and 6 lie completely on the quartz wall, as indicated in Figure 1. The remaining two spray cones impinge mainly on the metal frame next to the quartz window. A pair of electrodes below the injector (see Fig. 1) ignites the fuel/air-mixture 0.7 ms after the end of injection (EOI). Therefore, the experiment employs a combination of a spray-guided and wall-guided direct-injection strategy. The latter is known to potentially lead to increased soot formation [30].

![Figure 1](image1.png)

**Figure 1**: (left) Optically accessible test section of wind tunnel with injector and spark electrodes. (right) Numbering of the six fuel films.

Table 1 gives an overview of the operating conditions in the experiments. The air, wall, and injector temperatures cannot be controlled independently but follow the air temperature as given in Table 1.

| Table 1: Operating conditions. |
|---------------------------------|
| Fuel | Iso-octane |
| Injector | Six holes |
| Rail pressure | 100 bar |
| Injector temperature | 361 K |
| Injection duration | 1 ms (9.3 mg) |
| Air temperature | 381 K |
| Air flow velocity | 1.8 m/s |
| Back pressure | 1 bar |
| Quartz wall temperature | 352 K |

**Optical layout**
The layout for the simultaneous visualization of PAH and soot is shown in Figure 2. A laser sheet at 532 nm was put into the test section to excite laser-induced fluorescence of PAH. A second laser sheet at 1064 nm was put into the test section to excite laser-induced incandescence of soot. The laser sheets were aligned in a way that they intersect with fuel films 2 and 6. Behind the output of the 532 nm laser, a Pellin-Broca prism separated remaining infrared light from the 532 nm beam and deflected the beam about 90°. A mirror reflected the 532 nm laser towards the test section. Two mirrors directed the 1064 nm beam towards the test section in a way that it had the same pathway as the 532 nm laser. A positive cylindrical lens (f = 400 mm) focused the laser beams horizontally. A negative cylindrical lens (f = -25 mm) expanded the beams vertically into light sheets. An aperture clipped the light sheets vertically so that the two laser sheets opened up a two-dimensional plane with a height of 80 mm and a
thickness of 0.5 mm. A beam splitter, BS509 (Semrock, Beamsplitter HC BS 509 imaging), transmitting light with a wavelength above 509 nm and reflecting light with a wavelength below, was used to separate PAH LIF from soot LII. An intensified CCD camera, equipped with a lens (Nikon, \( f = 50 \) mm, \( f/1.8 \)) and a bandpass filter, BP 435/40 (Semrock, 435/40 BrightLine HC), captured soot LII. The gate of the intensifier was set to 60 ns, starting with the laser pulse. A second intensified CCD camera, equipped with a lens (Nikon, \( f = 50 \) mm, \( f/1.8 \)) and a bandpass filter (Schott, OG550), captured the red-shifted PAH LIF. Here, the gate of the intensifier was set to 30 ns, also starting with the laser pulse. The short gating suppressed the interference from natural flame luminosity. The temporal delay between the two laser pulses was 500 ns so that a quasi-simultaneous visualization was performed. The projected pixel size was 0.14 mm/pixel. The time between two images was 2.5 seconds. The laser fluences were set to 0.5 and 0.008 J/cm² for the 1064 and 532 nm laser pulses, respectively.

For preliminary measurements, the test section was replaced by a Santoro burner, providing a stationary sooting laminar coflow flame. First, the dependence of the LII signal versus the laser fluence was investigated. Second, the applicability of the measurement technique to simultaneously visualize PAH and soot was successfully tested. It consists of two concentric nozzles. The inner nozzle has an inner diameter of 10 mm and was fed with a volume flow of 0.231 sl/min of ethylene. The outer nozzle is a honeycomb that provided an air co-flow with a volume flow of 43 sl/min.

**Preliminary measurements**

Figure 3 shows ensemble average images of 1500 single shots of PAH LIF (left), excited with 532 nm, and of soot LII (center), excited with 1064 nm. Comparing these two images, shows that soot LII is also excited by the 532 nm laser. This is indicated by the spatial overlap of the signals in the LIF and LII images in the annular regions of the flame between a HAB of 30 and 50 mm and at HABs above 45 mm. In contrast to the LIF image, the LII image does not show signal in the hollow cone region below 45 mm, where the LIF image indicates signal. This is also shown by the single shot on the right hand side of Figure 3, showing soot LII and PAH LIF, simultaneously excited. The edges of the PAH LIF cone are covered by regions of high soot LII signal, indicating the nucleation region where soot forms from PAH.

**Figure 2:** Optical layout for PAH LIF and soot LII.
Figure 3: Ensemble average images of 1500 single shots of (left) PAH LIF and (center) soot LII. (right) Overlay of simultaneous soot LII and PAH LIF.

Figure 4 shows the PAH LIF and soot LII signals along the centerline with increasing HAB in the laminar diffusion flame of the Santoro burner. The LII signal starts to significantly increase at a HAB of about 30 mm and peaks at about 50 mm. The decrease in the LII signal is attributed to oxidation of soot, a decreasing temperature of soot particles, and decreasing laser fluence towards the wing of the Gaussian sheet. Surprisingly, the LIF signal peaks at around 40 mm, in a region where the LII signal starts to significantly increase. This indicates that the nucleation rate of soot becomes larger than the formation rate of large PAH. Also Santoro et al. found the soot volume fraction to strongly increase at a HAB of 37.6 mm for similar flow conditions and reported the initial of the oxidation region at a HAB of 50 mm. [31]. Smyth et al. also observed the maximum PAH LIF at a HAB where the soot LII starts to increase in an ethylene diffusion flame [28]. That indicates that PAH participate in soot production, either in soot inception or later as a growth species. Smyth et al. also observed that soot inception occurs in an interface between the PAH LIF in the fuel rich region. Recently, Hayashi et al. also found PAH and soot spatially well separated from each other in instantaneous measurements in a pulverized coal flame. They state that PAH mostly form in the premixed flame structure region while soot is present in the diffusion flame region [32].

Figure 4: LII and LIF signals along the centerline with increasing HAB.

Results
Figure 5 shows single shots of PAH LIF (green) and soot LII (red), simultaneously excited in the optically accessible test section of the flow facility at different times after start of injection. The
images are normalized to the global maximum signals of PAH LIF and soot LII within this case. The fuel films are indicated by the purple dots on the right hand side of the images. Film 2, in the top, has an initial mass of about 0.15 mg and has evaporated completely at about 80 ms after start of injection (aSOI). Film 6 has an initial mass of 0.37 mg and has evaporated completely at 125 ms aSOI. The fuel-film evaporation was measured with tracer laser-induced fluorescence, as described in [33].

**Figure 5:** Single shots of PAH LIF and soot LII images for the reference case from individual combustion events.

The quartz wall is indicated by the gray stripe on the right. The images show that PAH and soot mostly form in spatially well separated regions from each other. Both are detected in a shape of sharp filaments while the region of PAH appears more blurred than the one of soot. At 27 ms aSOI, soot is detected around the spark electrodes which stems from both the evaporating fuel film in the top and liquid fuel that adheres to the spark electrodes. At the same time, slightly above the fuel film in the bottom, PAH are detected close to the wall while no soot is adjacent. At 36 ms aSOI, soot and PAH are detected in adjacent curved regions, stretching out along the flow direction, which clearly shows the transition from PAH to soot. In the first single shot at 36 ms aSOI, a slight overlap of the two signal regions is present. In the second image, a dark region appears between the two species, indicating the nucleation zone. At 45 ms aSOI, PAH and soot mostly form in the top near film 2.

Corresponding to that, Figure 6 shows a sequence of a single combustion event in the same field of view, captured with a high-speed color camera at different times aSOI.

**Figure 6:** Sequence of high-speed images of natural soot luminosity, Mie scattered LED light from the spray, and chemiluminescence of the flame front.
The images are normalized to the full well capacity of the sensor, to 4095 counts. The Mie scattered light of an LED visualizes the penetration of the spray through the test section at the end of injection, at 1 ms aSOI. It also indicates where the fuel impinges on the wall and forms the liquid films 2 and 6. At about 1.7 ms aSOI the spark ignites the fuel/air-mixture and the turbulent flame front begins to propagate through the test section as indicated by the chemiluminescence signal at 5 ms aSOI. The turbulent flame front has completely propagated through the test section at 12 ms aSOI. A first soot pocket is detected slightly below film 2 at 9 and 12 ms aSOI. This soot most presumably results from inhomogeneities around both the wetted spark electrodes and film 2 on the quartz wall. The soot luminosity in the top becomes most intense at 18 ms aSOI and extinguishes with further progress in time while at the same time soot inception starts in the vicinity of film 6 in the bottom. From 30 to 48 ms aSOI soot is detected in various, spatially separated, regions and always close to the quartz wall. It needs to be taken into account that, in contrast to the laser-based measurements, the signal is integrated along the line of sight so that it also contains contributions from films 3 and 5.

Figure 7 shows ensemble average images of PAH LIF and soot LII. Soot is first detected in the top around the spark electrodes at 17 and 21 ms aSOI. Almost no PAH are detected at that times. At 24 ms aSOI, strong PAH LIF signal is detected in the bottom of the test section, near film 6. Here, very few soot is detected adjacent. The PAH LIF layer extends further to the bottom, covering film 6, at 27 ms aSOI. Soot filaments arise near this PAH layer. Also a layer of PAH with much weaker intensity is found near film 2. At the same time, soot LII in the top of the test section is becoming more intense.

![Figure 7: Ensemble average images of 200 single shots of PAH LIF and soot LII.](image)

Apparantly, the flow within the facility transports the PAH cloud from the bottom towards the top at 33 ms aSOI. Here, broadened clouds of PAH LIF and soot LII are detected next to each other, originating from the wall. At 36 ms aSOI, both signals become weaker and seem to stretch out from the wall towards the injector. Some soot is transported out of the test section at 36 ms aSOI, as indicated by the LII signal downstream of film 6. Both signals become weaker with further progress in time. This is either because both species are transported out of the test section or light-sheet plane or they are getting oxidized. At 51 ms aSOI, no soot is detected anymore and solely PAH LIF is detected near film 2. Most probably, temperatures are not high enough to form soot from PAH.

Ensemble average images of combustion luminosity, shown in Figure 8, visualize chemiluminescence, indicated in blue, and soot, indicated in red. Strong chemiluminescence signal
covers the field of view at 9 ms aSOI, indicating high temperatures in those regions. Consistent with
the results from Figure 7, first soot luminosity is detected in the top region, near film 2, at 15 ms aSOI.
Most probably, the high temperatures cause the fast soot formation in the top of the test section
without the presence of PAH. In contrast to Figure 7, significant soot is also found in the bottom near
film 6 at 18 ms aSOI. The maximum soot luminosity is detected, in good agreement with results from
soot LII, at 33 ms aSOI. The soot luminosity covers, compared to the LII signal, a larger area and
exhibits much less spatial intermittency. Surprisingly, chemiluminescence signal, indicating the
oxidation of either fuel, PAH, or soot, is found near film 6 throughout all images in Figure 8. In a very
close region, slightly upstream, strong PAH LIF signal arises between 24 and 33 ms aSOI. In good
agreement with the visualization of PAH LIF and soot LII, the sooting regions from the top and the
bottom merge together between films 2 and 6, starting at 33 ms aSOI. No soot LII or PAH LIF signal
is detected in the chemiluminescence region. With further progress in time, soot luminosity further
diminishes and the chemiluminescence increases, indicating enhanced oxidation. According to Figures
7 and 8, very little soot leaves the test section. At 51 ms aSOI, very little soot luminosity is detected in
the test section, indicating that most of the soot was oxidized.

![Figure 8: Ensemble average images of natural soot luminosity and chemiluminescence of the flame front.](image)

Conclusions
A measurement technique was developed to simultaneously visualize laser-induced fluorescence of
PAH, excited at 532 nm, and laser-induced incandescence of soot, excited at 1064 nm. In a first part of
this work the technique was applied on a sooting laminar coflow flame of a Santoro burner. Although
the laser fluence of the 532 nm laser was 62 times lower than the one from the 1064 nm laser, soot LII
was excited by the 532 nm laser in the flame. A comparison between the images, excited at 532 and
1064 nm, allowed for the discrimination between soot and PAH since the latter shows solely soot.
PAH LIF is detected in the laminar flame in a hollow cone region. The edges of the PAH LIF cone are
covered by regions of high soot LII signal, indicating the nucleation region where PAH turn into soot.
The PAH LIF signal peaks at a HAB of 40 mm where the LII signal strongly increases, indicating the
transition of PAH to soot.

In the second part, the measurement technique was applied to the optically accessible test
section of a wind tunnel to visualize soot formation near evaporating fuel films. Additionally, high-
speed color combustion-imaging was used to visualize chemiluminescence and natural soot
luminosity. Single shots show that PAH and soot mostly form in spatially well separated regions and
close to the fuel films. The early formation of soot without the presence of PAH in the top of the test
section occurs most probably fast due to high temperatures, as indicated by strong chemiluminescence
signal in this region. At later times aSOI, strong PAH formation occurs in the bottom of the test section, close to film 6 while very few soot is detected in this region. Most presumably, the temperature is insufficient to complete the transition of PAH to soot. With further progress in time the flow merges PAH from the bottom with soot from the top between the two fuel films and PAH LII and soot LII become strongest. Chemiluminescence signal can be found constantly in a region below PAH and soot, indicating the oxidation of the species being transported downstream. Images of soot LII and natural soot luminosity visualize soot in similar regions at different times after start of injection. Soot LII appears with a very high spatial intermittency while PAH form a more continuous cloud.

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