

3 Pyrene-LIF Thermometry of Early Soot Formation Region in a Diesel Spray Flame

Tetsuya AIZAWA and Hidenori KOSAKA

Tokyo Institute of Technology

2-12-1 Ohokayama, Meguro-ku, Tokyo 152-8552, Japan

In order to investigate early soot formation process in diesel combustion, spectral analysis and optical thermometry of early soot formation region in a transient spray flame under diesel-like conditions was attempted via laser-induced fluorescence (LIF) from pyrene ($C_{16}H_{10}$) doped in the fuel. Pyrene is known to exhibit a temperature dependent variation of LIF spectrum; the ratio of S_2/S_1 fluorescence yields, from the second/lowest excited singlet states, depends on temperature. In the present study, pyrene was doped in a model diesel fuel and the variation of LIF spectra from the pyrene in the spray flame were examined at different ambient temperatures, ambient oxygen concentrations, measurement positions and timings after start of fuel injection. The pyrene LIF spectra measured in spray flame up to ignition showed increase in the S_2/S_1 ratio of LIF intensity and shift of the peak wavelength of S_1 towards red as the time progresses after start of injection, showing that the temperature variation of the gas mixture up to ignition can be captured by this technique. The LIF spectra after ignition showed increase in emission intensity in longer wavelength region due to LIF from combustion products.

Keywords: Diesel Combustion, Optical Diagnostics, Soot Formation, Thermometry / Pyrene-LIF

1. INTRODUCTION

In the development of latest diesel engine systems under ever stringent particulate emission regulations, application of premixed compression ignition in partial-load operational ranges and various after treatment technologies has been examined. On the other hand, the potential of smokeless diesel spray combustion based on a variety of approaches, such as low temperature combustion^(1,2,3,4), high pressure fuel injection and high boost charging⁽⁵⁾, has been attracting a growing attention. In order to explore and enhance the potential of these emerging spray combustion technologies, detailed chemical and physical understanding of soot formation processes in the spray flame is becoming increasingly important. It is well known that general trend of soot and NOx emission from diesel combustion can be described by two important parameters, temperature and equivalence ratio. However, the soot reduction mechanism in the smokeless spray combustion has not fully been examined by directly measuring these two important parameters. Numerical simulation of particulate formation in diesel spray flame has extensively been performed using detailed chemical reaction schemes and a variety of particle inception models^(6,7,8,9). However, the particulate formation and oxidation models used in the numerical analysis are still emerging and not capable of fully describing the mechanism of smokeless spray combustion. Comprehensive experimental investigation is necessary to improve and validate the models and the simulated results.

In order to investigate the soot formation process in diesel spray flame, our group has recently conducted spectral measurements of laser-induced emission⁽¹⁰⁾ and simultaneous 2-D imaging of soot precursor and soot particles in a diesel spray flame using laser-induced fluorescence (LIF) and laser-induced incandescence (LII) techniques^(11,12). The measurement results revealed that the soot precursors are observed in the central fuel-rich region of the spray flame immediately after ignition and the soot particles gradually appear from the periphery of the soot precursor region and extend downstream. The LIF from the soot

precursors exhibited very broad spectra which are typical of polycyclic aromatic hydrocarbons (PAHs). The spectra changed from the broad LIF to gray-body emission due to LII from soot particles in the downstream region as the time progresses after ignition. These measurement results have considerably improved our understanding about the soot formation process in a diesel spray flame. However, temperature and equivalence ratio, which are considered to be the most important parameters for soot formation process, have not been measured in this central soot precursor region of the spray flame.

As a potential technique for simultaneous measurement of temperature and equivalence ratio in the early soot formation region in a diesel spray flame, we are investigating pyrene-LIF technique. Pyrene ($C_{16}H_{10}$) is known to exhibit temperature dependent variation of LIF spectrum known as "anomalous" fluorescence^(13,14). Peterson et al. used of this temperature dependent fluorescence of pyrene to measure the temperature of low pressure (up to 80 Torr) burner flames doped with vapor-phase pyrene^(15,16). Pyrene also has a high quantum efficiency for LIF, which will provide sufficient intensity of LIF for planar imaging measurement of fuel concentration. In addition, pyrene is one of the most abundant PAH components or soot precursors in flames. An attempt to investigate how the pyrene doped in the fuel changes into soot particles in a diesel spray flame may provide us with information useful for further understanding of the soot formation process.

In this paper, in order to investigate early soot formation process in diesel combustion, spectral analysis and optical thermometry of early soot formation region in a transient spray flame in a rapid compression machine was attempted via LIF from pyrene doped in the fuel.

2. PYRENE LIF THERMOMETRY

It is known that fluorescence from polyatomic organic molecules such as PAHs generally follow Kasha's rule, where highly efficient internal conversion results in emission

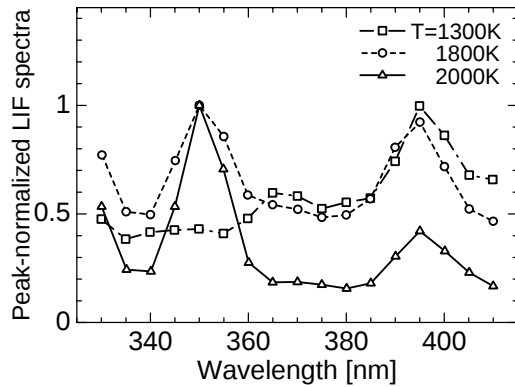


Fig.1 Pyrene LIF spectra measured in C_2H_4/O_2 flame at different temperatures (Ref.15)

originating from the lowest excited singlet state S_1 regardless of the excitation energy⁽¹⁷⁾. However, a couple of PAHs such as pyrene, azulene and its derivatives are known to have emission also from the S_2 state termed anomalous fluorescence. The ratio of S_2/S_1 fluorescence intensities has strong temperature and excitation energy dependence. Fig.1 shows the spectra of laser-induced fluorescence from doped pyrene in sub-atmospheric C_2H_4/O_2 flame taken by Peterson et al.⁽¹⁵⁾ Two prominent peaks around 350nm and 400nm corresponds to fluorescence from S_2 and S_1 bands, respectively. The ratio of S_2/S_1 fluorescence intensities varies with temperature.

The ratio of S_2/S_1 fluorescence yields at temperature T can theoretically be written by Eq.(1).

$$S_2 / S_1 = \frac{\int_0^\infty \frac{\rho_2(E + \Delta E)}{\rho_1(E + \Delta E)} \rho_0(E) \exp(-E/kT) dE}{\int_0^\infty \rho_0(E) \exp(-E/kT) dE} \quad \text{-----(1)}$$

where E is energies greater than the excitation energy, ΔE is the excitation energy in excess of the zero-order S_0-S_1 transition energy where S_0 denotes the electronic ground state, ρ_i is the density of vibrational levels for the i th electronic state. Fig.2 shows the temperature dependence of the S_2/S_1 fluorescence yields obtained by Eq.1 at the excitation wavelength of 309nm which was used in the present study. Peterson et al. used of this temperature dependence to measure the burner flame temperature. In the present study, the potential of this pyrene-LIF technique for temperature measurement in early soot formation region in a diesel spray flame is investigated.

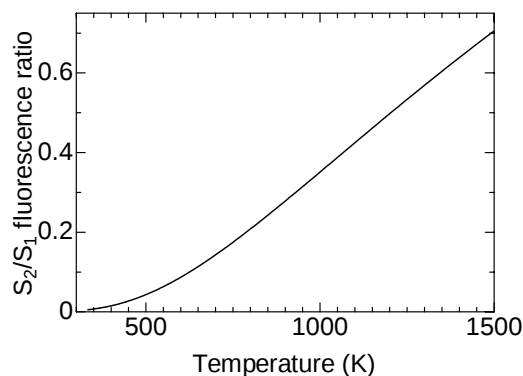


Fig2 Theoretically predicted temperature dependence of S_2/S_1 emission yield of pyrene

2. EXPERIMENTAL SETUP

Fig.3 shows the optical setup for pyrene-LIF spectral measurements in a transient spray flame. The 2nd harmonic of a pulsed Nd:YAG laser (450mJ) pumped a dye laser (Lambda Physik Scanmate) and generated excitation laser pulses (309nm, 5mJ) for LIF measurements. The laser pulse was gradually focused by a quartz spherical lens ($f=750$ mm) and passed through the central axis of the spray flame achieved in an optically accessible rapid compression machine (RCM). The diameter of the laser beam was approximately 5mm at the measurement location. The LIF from the spray flame was collected by two spherical quartz lenses (F0.57) and focused onto an entrance slit of a spectrometer (Solar TII, focal length=25cm) with an ICCD camera (Andor iStar). The gate width of the ICCD camera was set to 100ns, which was short enough to eliminate natural emission from the spray flame. The spatial resolution of the spectral detection system calculated by the lenses' F-number and the slit width was approximately 4mm x 0.1mm, along and perpendicular to the central axis of the spray flame, respectively. Long-pass filters (cutoff wavelength 340nm) were used to eliminate scattered laser light from soot particles. The measurements were conducted mainly at 30mm to 50mm downstream from the nozzle orifice along the central axis of the spray flame, where LIF from soot precursors were observed in previous imaging and spectral measurements in the spray flame^(10,11,12) under identical conditions. Pyrene (Wako Chemical, 98% purity) was dissolved in a model diesel fuel (0-solvent) at 1%wt concentration which was required to generate sufficient LIF for spectral measurements in the spray flame.

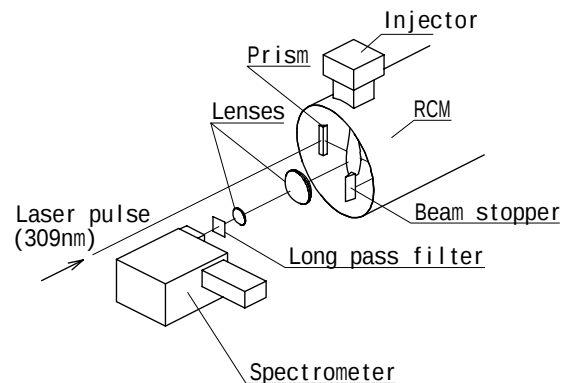


Fig.3 Experimental setup of pyrene-LIF spectral measurements in diesel spray flame

Table.1 Experimental conditions

Surrounding conditions		
Pressure : P_a		2.8MPa
Temperature : T_a		620K, 710K, 810K
Oxygen concentration : X_{O_2}		0% ~ 21%
Ignition delay : τ		0.8ms ~ 3.8ms
Measurement parameter		
Measurement timing		0.6ms ~ 3.0ms
Axial location of detection		30mm, 40mm, 50mm, 80mm
Injection conditions		
Nozzle diameter: d_0		0.15mm
L / d_0		4
Fuel	0-Solvent (n- $C_{12} \sim C_{14}$)	99.0 wt%
	Pyrene ($C_{16}H_{10}$)	1.0 wt%
Injection amount : M_f		16mg
Injection pressure : ΔP_{inj}		65MPa
Injection duration : t_{inj}		3.7ms

The rapid compression machine (RCM) used in this study has a bore of 200mm, a stroke of 560mm and a compression ratio of 14.7⁽¹⁸⁾. The optically accessible combustion chamber is pancake type with a diameter of 196mm and thickness of 40mm at TDC. An electronically controlled and hydraulically actuated single-shot unit injector⁽¹⁹⁾ was used for fuel injection. The experimental conditions are listed in Table 1.

3. RESULTS AND DISCUSSION

4.1 PYRENE LIF MEASUREMENTS IN EVAPORATING SPRAY

Pyrene LIF spectra were measured in non-combusting evaporating spray in nitrogen atmosphere at diesel-like temperature and pressure in a rapid compression machine. Reference spectra of pyrene LIF without any possible effect of chemical reaction on pyrene molecules and without spectral interference due to LIF from combustion products were obtained. Variation of pyrene LIF spectra with ambient temperature was also observed. Fig.4 shows pyrene LIF spectra measured in non-combusting evaporating spray at different ambient temperatures, distances from the nozzle orifice, and measurement timings after start of fuel injection. The spectra were normalized by the peak intensity. It should be noted that the intensity of background emission, which was mainly LIF from fuel impurities and measured by using the base fuel without dissolved pyrene, was less than 1% of the peak intensity of pyrene LIF and negligible. Each spectral plot shown below is an average spectrum of 3 to 6 measurements. The spectra have been corrected by spectral transmission of long-pass filters and spectral sensitivity of the spectrometer. The temperatures shown here are thermodynamic gas temperatures calculated for the entire bulk mixture. Temperatures measured by Pt wire resistance thermometer at the center of the combustion chamber were higher than the bulk gas

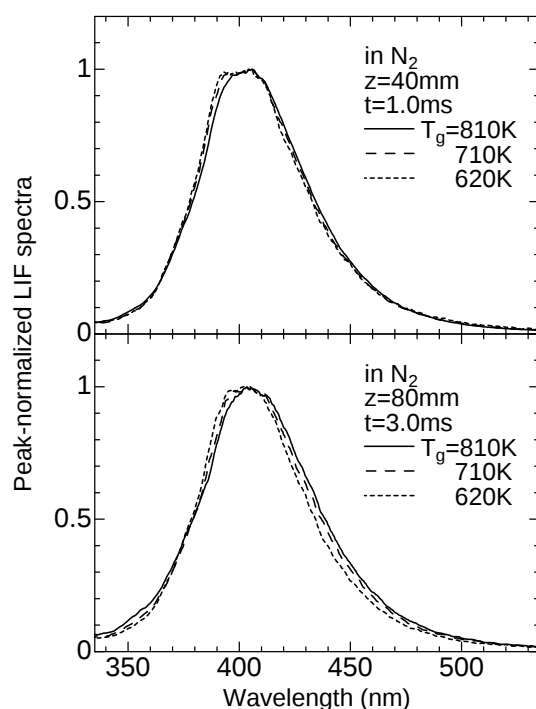


Fig.4 Pyrene LIF spectra measured in non-combusting evaporating spray in nitrogen atmosphere at different ambient temperatures, measurement positions and timings

temperature by 50 to 80K.

A comparison between Fig.1 and 4 shows that the pyrene LIF spectra in sub-atmospheric burner flame shown in Fig.1 exhibit two prominent peaks around 350nm (S_2 band) and 400nm (S_1 band) especially at higher temperatures, while the spectra in evaporating spray show only one broad peak around 400nm. This difference is mainly due to temperature difference between Fig.1 and 4. The relative fluorescence yield of S_2 band increases at higher temperature and the temperature in the evaporating spray was too low for the S_2 band fluorescence to be clearly distinguished in the spectra. However, the spectra shown in Fig.4 were in general agreement with fluorescence spectra of pyrene vapor measured at temperatures under 1000K using an electrically heated optical cell, atmospheric burner flames and a shock tube in other references^(20,21,22).

The spectra measured at different temperatures shown in Fig.4 indicate that the fluorescence intensity around 350nm corresponding to S_2 band slightly increases and the intensity around 400nm corresponding to S_1 band slightly decreases, with increase in ambient temperature. In addition, the fluorescence observed in longer wavelength region beyond 400nm broadens towards red with increase in ambient temperature. Fig.4 also indicates that these variation of spectral profile with increase in ambient temperature is more recognizable in the spectra measured at upstream position in the spray ($z=40$ mm) at earlier timing ($t=1.0$ ms) after start of fuel injection than the spectra measured at downstream position ($z=80$ mm) at later timing ($t=3.0$ ms).

In order to see how the spectral profile is reflecting the above mentioned variation of S_2/S_1 fluorescence intensities with temperature, the temperature at the measurement location was estimated assuming that the S_1 band fluorescence intensity corresponds to the area of integrated spectral profile between 395nm and 420nm and the S_2 intensity corresponds to the area between 345nm and 370nm, and using the S_2/S_1 to temperature relation shown in Fig.2. The obtained S_2/S_1 ratios and the estimated temperatures are listed in Table 2 and 3. Temperatures for the downstream location ($z=80$ mm) were estimated to be higher than the upstream location ($z=40$ mm) by about 40K to 80K, which can be attributed to temperature increase in the spray due to mixing of fuel vapor with high temperature ambient nitrogen in the downstream location at the later timing.

Table 2. S_2/S_1 ratio and calculated temperature (in N_2 , $z=40$ mm, $t=1.0$ ms)

Ambient temperature	S_2/S_1 ratio	Calculated temperature
620K	0.150	710K
710K	0.147	706K
820K	0.150	710K

Table 3. S_2/S_1 ratio and calculated temperature (in N_2 , $z=80$ mm, $t=3.0$ ms)

Ambient temperature	S_2/S_1 ratio	Calculated temperature
620K	0.177	755K
710K	0.175	750K
820K	0.201	792K

4.2 PYRENE LIF MEASUREMENTS IN SPRAY FLAME

Pyrene LIF spectra were measured in a transient spray flame at different distances from nozzle orifice, timings after start of fuel injection, ambient temperatures, ambient oxygen concentrations in order to investigate the variation of spectral profile. Fig.5 shows the pyrene LIF spectra measured in the

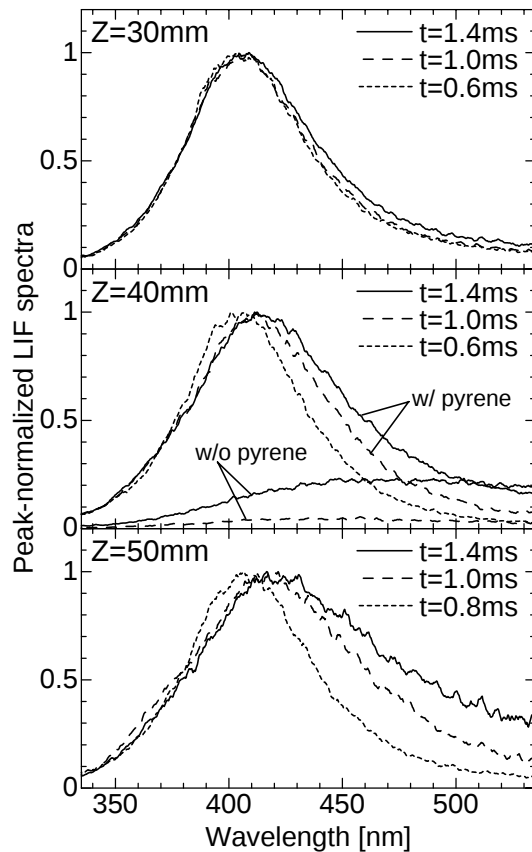


Fig.5 Pyrene LIF spectra measured in spray flame at different measurement positions and timings (O_2 21%, $T_g=710K$, ignition delay $\tau=1.0ms$)

spray flame at different distances from nozzle orifice and timings after start of injection. Ambient gas is air (O_2 21%) and bulk temperature is 710K. The ignition delay detected by pressure increase due to heat release was 1.0ms. Spectra of background flame luminescence have been subtracted. The background correction was below few percent of the peak LIF intensity. Noticeable differences were not observed in ignition delays and heat release curves between the base fuel and the pyrene-doped fuel.

In Fig.5, before ignition ($t=0.6ms$ and $0.8ms$), the measured spectra are almost identical between different distances from nozzle orifice. These spectra are also very similar to the spectra measured in evaporating spray shown in Fig.4. On and after the ignition ($t=1.0ms$ and $1.4ms$), the spectra indicate that the intensity of the S_2 band fluorescence around 350nm slightly increases and the S_1 band peak around 400nm shifts towards red in downstream positions and at later timings. In addition, the intensity of fluorescence observed in longer wavelength region beyond 420nm increases in downstream positions and at later timings. This increase in the fluorescence in longer wavelength region is due to background LIF from soot precursors produced in the spray flame. The middle plot in Fig.5 at $z=40mm$ shows LIF spectra of combustion-generated soot precursors measured using the base fuel without pyrene. Vertical scale of the spectra of these base-fuel LIF corresponds to their pyrene-doped counterparts. The contribution of the background soot precursor LIF is almost negligible at the ignition timing ($t=1.0ms$). However, after ignition ($t=1.4ms$), the contribution significantly increases especially in longer wavelength region.

The S_2/S_1 ratio and the temperature at different measurement locations were estimated in the same manner as the above mentioned evaporating spray cases and the calculated

results are listed in Table 4. The estimated temperatures in Table 4 are higher than the non-combusting evaporation spray cases shown in Table 2 and 3. The temperatures estimated for downstream locations are higher than the ones for upstream locations, except for the case in $z=50mm$ and $t=1.4ms$ which suffers significant interference from the background LIF. This general trend of increasing temperature in downstream locations demonstrates that the temperature increase due to the progress of chemical reaction in the spray can be captured by this measurement technique.

Table 4. S_2/S_1 ratio and calculated temperature at different measurement locations (O_2 21%, $T_g=710K$, ignition delay $\tau=1.0ms$)

Meas. location	Meas. timing	S_2/S_1 ratio	Calculated temperature
Z=30mm	0.6ms	0.200	786K
	1.0ms	0.214	812K
	1.4ms	0.217	812K
Z=40mm	0.6ms	0.222	819K
	1.0ms	0.238	848K
	1.4ms	0.245	855K
Z=50mm	0.8ms	0.219	819K
	1.0ms	0.281	903K
	1.4ms	0.244	855K

Fig.6 shows the pyrene LIF spectra measured in the spray flame at different ambient temperatures and timings after start of injection. Ambient gas is air (O_2 21%) and the measurement

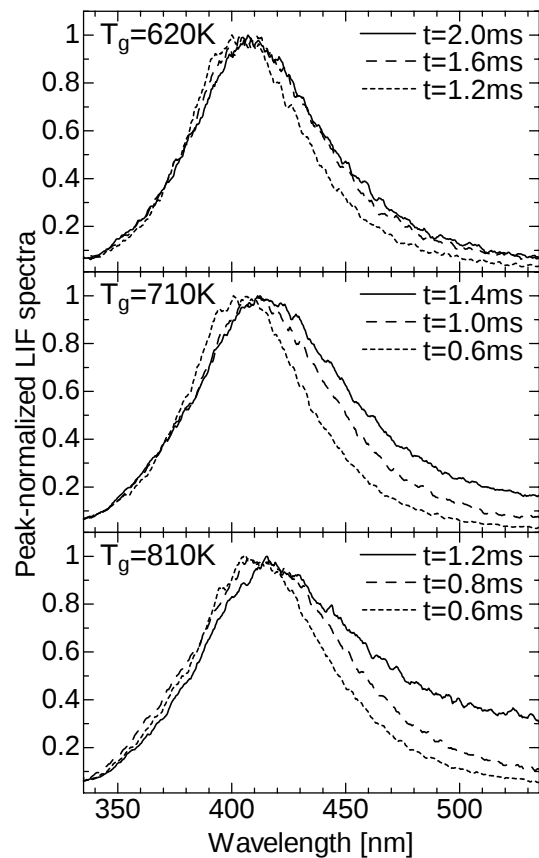


Fig.6 Pyrene LIF spectra measured in spray flame at different ambient temperatures and measurement timings ($O_2=21\%$, $z=40mm$, ignition delay $\tau=1.6ms$, $1.0ms$, $0.6ms$ for $T_g=620K$, $710K$, $810K$, respectively)

location is 40mm downstream from the nozzle orifice. The ignition delay varied from 0.8ms to 1.6ms depending on the ambient temperature. The spectra were measured at three different timings for each ambient temperature cases, 0.4ms before ignition, at the ignition and 0.4ms after ignition, except for the $T_g=810\text{K}$ case where 0.4ms before ignition was too early for the LIF from the spray to be detected at the measurement location. The general trend of the variation of the LIF spectral profiles observed in Fig.6 is similar to the trend observed in the cases for different distances from nozzle orifice shown in Fig.5. The intensity of the S_2 band fluorescence around 350nm slightly increases and the S_1 band peak around 400nm shifts towards red at higher ambient temperatures and at later timings. The intensity of fluorescence observed in longer wavelength region beyond 420nm increases at higher ambient temperatures and at later timings due to background LIF from soot precursors produced in the spray flame.

The estimated S_2/S_1 ratios and the temperatures for different ambient temperatures are listed in Table 5. The temperatures estimated for higher ambient temperatures and later measurement timings are higher, except for the case in $T_g=810\text{K}$ and $t=1.2\text{ms}$ with significant interference from the background LIF. These results demonstrate that the temperature increase due to higher ambient temperature and the progress of chemical reaction in the spray can be captured by this measurement technique.

Table 5. S_2/S_1 ratio and calculated temperature at different ambient temperatures

Ambient temperature	Meas. timing	S_2/S_1 ratio	Calculated temperature
620K	1.2ms	0.209	799K
	1.6ms	0.224	826K
	2.0ms	0.229	833K
710K	0.6ms	0.222	819K
	1.0ms	0.238	848K
	1.4ms	0.245	855K
810K	0.6ms	0.228	833K
	0.8ms	0.264	879K
	1.2ms	0.219	819K

Fig.7 shows the pyrene LIF spectra measured in the spray flame at different ambient oxygen concentrations (21% to 0%). Ambient temperature is 710K and the measurement location is 40mm downstream from the nozzle orifice. The ignition delay detected by pressure rise was heavily dependent on the ambient oxygen concentration and varied from 1.0ms for $O_2=21\%$ to

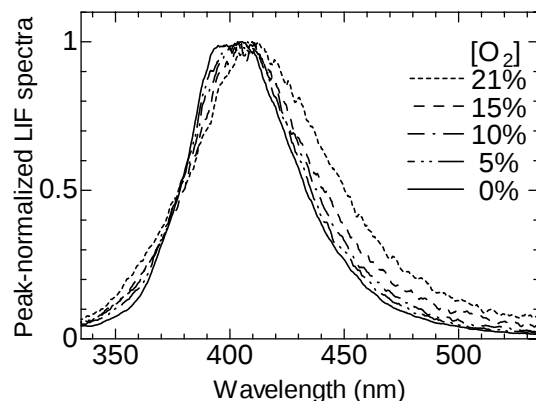


Fig.7 Pyrene LIF spectra measured in spray flame at different ambient oxygen concentrations ($t=1.0\text{ms}$, $z=40\text{mm}$, $T_g=710\text{K}$)

3.8ms for $O_2=5\%$. The measurement timing, on the other hand, was fixed to 1.0ms after start of fuel injection. Therefore, all spectra shown in Fig.7 were measured before ignition. The spectra shown in Fig.7 indicate that the intensity of the S_2 band fluorescence around 350nm increases with increase in ambient oxygen concentration, which might be attributed to temperature increase due to the progress of combustion reaction in the spray. It should be mentioned that Ossler et al. have reported that the LIF spectral profile of pyrene vapor at atmospheric pressure and moderately high temperature (430K) has some sensitivity to quenching by oxygen molecules⁽²³⁾. Therefore, the changes in the spectral profile shown in Fig.7 might not be purely due to temperature, but also due to oxygen quenching. The spectra shown in Fig.7 does not exhibit the intensity increase in longer wavelength region due to background LIF from soot precursors as shown in Fig.5 and 6. However, the S_1 band peak around 400nm still indicates the shift towards red at higher ambient oxygen concentrations. The estimated S_2/S_1 ratios and the temperatures for different ambient oxygen concentrations are listed in Table 6. The temperatures estimated for higher ambient oxygen concentrations are higher.

Table 6. S_2/S_1 ratio and calculated temperature at different ambient oxygen concentrations

O_2 concentration	S_2/S_1 ratio	Calculated temperature
0%	0.147	706K
5%	0.173	749K
10%	0.192	773K
15%	0.200	786K
21%	0.238	848K

The measurement results of the pyrene LIF spectra in a transient spray flame shown above can be summarized as follows. In all cases with different measurement locations, measurement timings after start of injection, ambient temperatures, and ambient oxygen concentrations, the intensity of the S_2 band fluorescence around 350nm increases and the S_1 band peak around 400nm shifts towards red, with increase in temperature due to the progress of chemical reaction in the spray. After ignition, the intensity of fluorescence in longer wavelength region increases due to background LIF from soot precursors produced in the spray flame.

The estimation procedure for S_2/S_1 ratio of fluorescence yield and temperature employed in the present paper is the first approximation and has to be further improved, in which not only the spectral range of each band, but also spectral overlapping of two bands have to be considered. However, it is interesting that significant interference from the background LIF from soot precursors are already observed in the spray as shown above in the spectra, despite the estimated temperatures below 1000K. Future work necessary to further investigate the potential of the pyrene-LIF technique for thermometry in soot formation region in a diesel spray flame includes development of temperature estimation procedure in which the interference from the background LIF from soot precursors can be reduced, and calibration of spectral profiles of pyrene LIF to temperature using a shock tube⁽²²⁾. It should also be considered to use a shorter excitation wavelength, because S_2 band LIF intensity is significantly enhanced by using a shorter excitation wavelength.

5. CONCLUSION

In order to investigate early soot formation process in diesel combustion, spectral analysis and optical thermometry of early soot formation region in a transient spray flame in a rapid

compression machine under diesel-like conditions was attempted via laser-induced fluorescence from pyrene doped in the fuel and the following results were obtained:

- (1) The pyrene LIF spectra measured in non-combusting evaporating sprays in nitrogen atmosphere at different ambient temperatures exhibited slight increase in the fluorescence intensity around 350nm corresponding to S_2 band and the fluorescence observed in longer wavelength region beyond 400nm broadened towards red with increase in ambient temperature.
- (2) The pyrene LIF spectra measured in transient spray flames at different distances from the nozzle orifice to the measurement location, measurement timings after start of injection, ambient temperatures, and ambient oxygen concentrations, exhibited increase in the intensity of the S_2 band fluorescence around 350nm and the S_1 band peak around 400nm shifted towards red, with increase in temperature due to the progress of chemical reaction in the spray. The spectra measured after ignition exhibited increase in the fluorescence intensity in longer wavelength region due to background LIF from soot precursors produced in the spray flame.

ACKNOWLEDGMENTS

The authors thank Toshio Douke of Tokyo Institute of Technology for his substantial experimental support and helpful discussions.

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