Joint Temperature and Soot-Volume-Fraction Measurements in Turbulent Meter-Scale Pool Fires

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The development of a combined dual-pump coherent anti-Stokes Raman scattering (CARS) and laser-induced incandescence (LII) instrument for the spatially resolved measurement of subgrid-scale temperature/soot data in liquid-fueled pool fires is discussed. Temperature pdfs obtained from the N₂ Q-branch CARS signal at the center of a 2-m-diameter toluene/methanol pool fire are summarized. A more detailed discussion of the recent development of a water-jacketed, fiber-optically coupled LII probe for in-fire soot-volume-fraction imaging is presented. Tomographically resolved laser-light-extinction characterization of the soot field in a fuel-rich premixed ethylene-air flame used for calibration of the LII technique is reported, and the performance of the LII-imaging system in the calibration flame is discussed. Two-dimensional LII images, which are representative of the spatially resolved, instantaneous soot-volume-fraction distributions in a 2-m-diameter toluene/methanol pool fire are provided, and a histogram of the LII signal that is representative of the pdf of the soot-volume-fraction fluctuations at the center of the fire are extracted from these in-fire imaging results. These data demonstrate the potential of the CARS and LII instruments to determine temperature and soot volume fraction in a sooting fire with high temporal and spatial resolution.

I. Introduction

LARGE-SCALE fires remain a dominant threat to safety and security for personnel and infrastructure. The danger posed is predominantly due to the immense thermal load, which is a result of radiative heat release from soot particles within the fire. Predictive simulation efforts are currently underway for use in fire-protection engineering and high-consequence risk-assessment calculations; however, the physics of soot radiative emission is dominated by processes that occur on the length scales of the individual flame sheets, which is below the computational grid size. In addition, soot volume fraction, unlike many other fire properties, does not correlate with the mixture fraction so that it is not readily modeled. Further development and refinement of the predictive models, therefore, require highly resolved, both temporally and spatially, experimental data for validation and verification of empirically based subgrid-scale radiative-emission source models. Traditionally, fire environments have been investigated using physical probes, such as thermocouples. However, the temporal and spatial resolution requirements for model development exceed physical probe capabilities and so optically based diagnostic techniques such as emission/absorption;¹ ³ coherent anti-Stokes Raman scattering (CARS);⁴ ⁵ tunable-diode-laser absorption spectroscopy;⁶ and laser-induced incandescence (LII), which exhibit the necessary resolution, are beginning to be utilized for fire research.

To meet the needs of the fire-modeling community, we are actively pursuing the development of optical-diagnostic tools for the spatially and temporally resolved measurement of the joint fluctuations of soot and temperature in turbulent fire plumes using dual-pump CARS and LII. For thermometry, CARS has a proven record as an accurate and powerful diagnostic in both laboratory combustion environments and in large-scale facilities.⁸⁻¹⁰ Large-scale, turbulent, sooting fires, however, exhibit challenges, which require a more sophisticated variant of the conventional CARS technique. The high degree of turbulent mixing present in large fires demands single-laser-shot spectral acquisition and the presence of appreciable amounts of soot may lead to both Swan-band and Raman-resonant spectral interferences from C₂ when using CARS with frequency degenerate pump beams.¹¹ Employment

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of broadband, dual-pump CARS\textsuperscript{12} permits single-shot measurements and allows the flexibility of a frequency tunable signal to remove spectral interferences from soot.\textsuperscript{11, 13, 14} For soot measurement, the only well-developed diagnostic technique available with the necessary temporal and spatial resolution is LII.\textsuperscript{15} This approach is based upon the continuum radiative emission of laser-heated soot particles entrained in the fire, which can be shown to be proportional to the total volume of soot in the measurement region.\textsuperscript{15-17} A laser of arbitrary frequency is used to heat soot particles within the probe volume to temperatures approaching the vaporization point near 4000-5000 K. At these temperatures, the emission from laser-heated soot is well in excess of background flame radiation, and the wavelength distribution of the emission from the laser-heated soot shifts from the infrared to the visible portion of the spectrum, permitting further background isolation based on spectral filtering. Once properly calibrated, the soot volume fraction can be readily determined. LII has been used for spatially resolved soot imaging in a variety of laboratory and practical combustion studies, as summarized in the review article by Santoro and Shaddix.\textsuperscript{15}

In this paper, we present the status of the development of a joint soot-volume-fraction/temperature diagnostic, which will couple broadband, dual-pump CARS thermometry with LII determination of the soot volume fraction. The CARS measurements presented have been discussed in detail elsewhere,\textsuperscript{4, 5} so we offer only a brief summary of the present state of our CARS thermometry capability here. A newly–developed, fiber-coupled LII instrument is then discussed and the present state of our soot measurement capability in meter-scale fires is reported.

II. Experimental

The data presented here have been acquired in the Sandia National Laboratories Fire Laboratory for Accreditation of Models and Experiments (FLAME) facility. This facility has the capability to produce well-controlled, reproducible, quiescent fires up to 3 m in base diameter using either liquid or gaseous fuels. A schematic of the facility is given in Fig. 1. The facility consists of a main fire bay, which is ~18 m in diameter and ~12 m in height with water-cooled walls. Air is supplied through the basement, where it is distributed around an annular vent. The air rises through the grated floor and is drawn horizontally across the ground plane, formed by a ~12-m-diameter annular steel apron, and into the fire by natural draft. Exhaust gases are vented through the roof of the fire bay to an electro-static precipitator, which removes any particulate before venting the exhaust gases to atmosphere. Adjacent to the main fire bay is the West Laser Lab, which houses the lasers and optical components required for the diagnostic measurements. For the data presented here 10% and 30% toluene in methanol, by volume, blended fuels were used, and the fires lasted 15-20 minutes, which allows for several thousand single-laser-shot measurements to be acquired.

The broadband, dual-pump CARS measurements require the use of three different laser systems and a diagram is given in Fig. 2 showing the experimental layout. An injection-seeded (bandwidth ~0.003 cm\textsuperscript{-1}), frequency-doubled, Q-switched Nd:YAG laser is operated at 10 Hz and produces ~1.7 J/pulse at 532 nm. This Nd:YAG laser serves as the pump for both a broadband and a narrowband, tunable dye laser, and provides one pump beam for the CARS process. The broadband dye laser emits the Stokes beam for the CARS process and uses a mixture of Rhodamine 640/610 in methanol to produce a beam with a bandwidth of ~225 cm\textsuperscript{-1} centered at 607 nm. The narrowband dye laser provides the second pump beam for the CARS process at ~560 nm and has a nominal, manufacturer-specified bandwidth of ~0.08 cm\textsuperscript{-1}. Telescopes are inserted in the dye laser paths to allow optimization of the laser-beam focusing at the measurement volume. The polarization vectors of the three beams are parallel and the energies of each beam for the data presented here are 50, 50 and 30 mJ/pulse for pump-1, pump-2 and Stokes beams, respectively. The three beams are aligned in the folded BOXCARS configuration before being input into the main fire bay. Inside the test bay (Fig. 3), the beams are directed to a set of optical housings which protect the optics and protrude into the fire to limit beam steering (housing separation ~0.7 m). A 1-m-focal-length, spherical lens in the first optical housing focuses and crosses the input beams ~1 m above the center of the fuel pan, resulting in a probe volume ~100-200 \( \mu \text{m} \) in diameter and ~10 mm long. The three input beams and the generated CARS signal are collected and collimated by a 1-m-focal-
length spherical lens in the second optical housing. A series of four dichroic mirrors separates the CARS signal from the pump and Stokes beams and a 100-mm-focal-length lens couples the signal into a 100-µm-diameter multimode optical fiber. An interference filter placed prior to the optical fiber removes any remaining pump or Stokes laser power while transmitting better than 80% of the CARS signal (λ~496 nm). The 30-m-long fiber couples the CARS signal into the 0.75-m spectrograph (50-µm slits), which is located in the West Laser Lab. A 1200 l/mm grating disperses the signal, which is magnified by a factor of 3.75 by a relay lens pair prior to focusing onto a back-illuminated, unintensified CCD detector (quantum efficiency ~95% at 496 nm). The nominal resolution of the detector is ~2.1 cm⁻¹. Previous work has indicated that the temperatures provided by our CARS instrument are accurate to within 2-3% and the precision lies within ±3-5%.⁴,⁵

Acquisition of LII data requires an additional laser and optical detection system, which has been recently installed at the FLAME facility, and is shown schematically in Figs. 2 and 3. A frequency-doubled, Q-switched Nd:YAG laser was operated at 10 Hz, with a maximum output energy of 440 mJ/pulse at 532 nm in approximately 10-ns-duration pulses. The output of this laser was apertured and relayed to the fire bay and directed to the first optical housing, which was described previously. Inside the optical housing a 1-m-focal-length cylindrical lens focuses the beam into a sheet with approximate dimensions 25 mm in height × 300-500 µm in thickness. Incandescence from laser-heated soot particles is collected normal to the laser sheet by a water-cooled optical probe. A schematic and digital photographs showing the LII probe components and their position within the test bay are given in Figs. 4 and 5. The axis of the water-jacketed probe, which houses the collection optics and optical fiber array, is oriented at 39° to the facility floor, allowing for the intensified CCD camera to be protected at the basement level. The camera, notch filter and output end of the fiber bundle are encased in a darkened enclosure to protect these instruments from room particulate and possible fuel spillage from the pan. The 1-m-focal-length, sheet-forming lens was rotated so that the laser sheet was normal to the axis of the collection optics.

The bulk of the LII collection optics are housed within a triple-walled, water-cooled, 2-m-long stainless-steel water jacket (~ 2GPM water flow; 101.6-mm O.D.; 50.8-mm I.D.). An

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**Figure 2. Diagram of the laser system. The recently added LII laser system is shown in the dashed box.**

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**Figure 3. Schematic of experiment in FLAME test bay. The ICCD camera is located in the basement of the test bay.**
uncooled, nitrogen-purged conical cap is mounted at the fire end of the water jacket to minimize soot formation on the probe windows and reduce the distance for absorption of the LII signal by soot in the intervening space between the laser sheet and collection optics. The collection-optics package is mounted inside the water-cooled tube and consists of an infrared reflecting window, an infrared absorbing window, and a relay-lens pair consisting of two 50.8-mm-diameter, 200-mm-focal-length, achromatic lenses. The optics package is currently configured to image at 1:1 magnification (depth of focus ~300-400 µm for a 25-µm feature) and relays the LII image to a 4.6-m-long optical fiber bundle with a 1-cm² face, 10-µm core optical fibers and a N.A. of 0.63. This unit-magnification imaging scheme provides a 1-cm square field of view in the object (fire) plane. The fiber bundle relays the LII signal to the detection system in the basement of the fire bay. A holographic notch filter centered at 532 nm with O.D. > 6, bandwidth < 350 cm⁻¹ and an out-of-band transmission of at least 75% is placed at the output end of the fiber bundle to remove any elastically scattered laser light. The signal is imaged using a 105-mm Nikon lens at f/2.8 onto an intensified CCD camera with a 512 × 512 array of 19 µm² pixels. The camera gate opens promptly with the arrival of the laser pulse and the gate width was typically set at 100 ns. The LII signal is detected broadband throughout the nominally 500-850 nm quantum response of the image intensifier with the exception of the 532-nm laser line. Broadband detection was used to maximize the LII signal coupled through the relatively slow f/4 imaging optics to the f/0.8 fiber bundle array, which then nominally overfills the f/2.8 Nikon lens on the ICCD detector. With this optical configuration, single-laser-shot LII detector counts observed during 2-m-diameter 30% toluene/70% methanol pool-fire burns averaged ~5000 counts above background with fluctuations observed throughout the 16-bit dynamic range of the ICCD.

Calibration of the LII measurements was performed using a fuel-rich (Φ = 3.1), premixed ethylene/air flame produced by a stainless-steel McKenna burner. The flow rates of the gas were 1.8 slpm ethylene, 8.2 slpm air and 1.0 slpm of N₂ co-flow with a burner cooling water flow rate of 0.14 slpm at a temperature of 19.4° C. A 101.6-mm-diameter × 12.7-mm-thick stainless-steel stabilizing plate was placed ~34 mm above the surface of the burner. The soot distribution within the fuel-rich flame was characterized using spatially resolved, light-extinction measurements, which were acquired using a HeNe laser. The laser was chopped, passed through a partial reflector so that a

Figure 4. Illustration of the LII optical probe.

Figure 5. Digital photographs showing position of LII optical probe relative to fuel pan, the uncooled cone at the LII probe tip, and the fiber-imaging bundle used to transmit the LII signal to the FLAME facility basement level.
reference of the baseline laser intensity could be acquired, and then focused with a 500-mm focal length lens into the calibration flame. Two large-area photodiodes, paired with 633-nm bandpass filters, were used to monitor the reference and signal intensities. The outputs of the photodiodes were detected using lock-in amplifiers, and the resultant lock-in signals were captured on a digital oscilloscope. The extinction measurements were acquired in horizontal scans with 1-mm spatial increments and averaged for 10 seconds. Multiple horizontal scans were acquired at heights above the burner ranging from 11-27 mm at 2 mm vertical increments. The path-integrated light-extinction data were tomographically inverted to yield the spatially resolved soot-extinction coefficient, $k_c$, using the three-point Abel algorithm advocated by Dasch.\textsuperscript{18} The $k_c$ data can be related to the soot volume fraction, $f_v$, by,

$$k_c = \frac{K_c f_v}{\lambda}$$

where $\lambda$ is the laser wavelength, and $K_c$ is the dimensionless soot extinction coefficient, which is a function of the soot refractive index. Reported values of $K_c$ in the literature vary by about a factor of 3, with more recent data from both postflame and in-flame soot in the range of 7 to 10 for a variety of fuels in laminar and turbulent flames.\textsuperscript{19, 20} We believe the recent evidence for these high $K_c$ values to be quite convincing and have employed a value of $K_c = 9$ here. Earlier measurements of the soot refractive index\textsuperscript{20, 21} are commonly used for interpretation of light-extinction data, where the inferred soot volume fractions will be higher by as much as a factor of 3.

### III. Results and Discussion

#### A. Summary of CARS Pool-Fire Thermometry Results

Our diagnostic systems have not yet reached the stage of development where simultaneous CARS/LII measurements can be acquired. However, both CARS and LII signals have been independently acquired from 2-m-diameter sooting, liquid-fueled pool fires. All N\textsubscript{2} Q-branch CARS and LII results to be shown here have been acquired at a measurement point that is nominally 1-m above the center of the 2-m-diameter liquid fuel pan using blended toluene/methanol fuels. Shown in Figs. 6 and 7, are sample single-laser-shot CARS spectra and the temperature pdfs for two 10% toluene/90% methanol burns. The sample spectra were fit using the Sandia National Laboratories CARSFT code.\textsuperscript{22} For this data set, the baseline adjust, temperature and N\textsubscript{2} mole fraction were treated as fitting parameters. Throughout the temperature range exhibited by the fire, the routine produces fits of quality similar to those displayed in Fig. 6 for the extraction of temperature from the N\textsubscript{2}-containing portions of the spectrum only. At temperatures below 1000 K there is a slight systematic overestimation of the calculated intensity on the right-side of the N\textsubscript{2} peak, which is more evident in the lower trace (blue) representing the difference between the experimental and calculated spectra. This is attributed to difficulty in measuring the instrument function, which is convolved with the theoretical CARS susceptibility. The mean and

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**Figure 6. Sample CARS spectra from 10% toluene/methanol burns.**
RMS temperatures for the two 10% toluene/90% methanol burns are 1140 ± 350 K and 1151 ± 402 K. The width of the temperature pdf illustrates the widely-varying local gas temperature within the fire plume as a result of large-scale turbulent “puffing” motions characteristic of buoyancy driven plumes and pool fires, which results in a gas composition within the probe volume that ranges from pockets of cold, unburned, gases to hot, post-combustion gases.

The average soot volume fraction in the fire plume is estimated to be of order 10⁻⁷ based upon the smoke yields of the blended methanol and toluene fuels. This degree of soot loading is not large enough so that the CARS thermometry measurements are significantly impacted by the optical thickness of the fire. Additionally, using the dual-pump CARS configuration effectively removes any spectral interference from C₂ typically found in CARS investigations of sooting flames using frequency-degenerate pump beams.

The temperature pdfs shown in Fig. 7 display similar mean temperatures for two repeat burns, with a 13% difference in the width of the pdf, accompanied by a shape difference on the low-temperature side.

**B. LII Calibration Measurements in the McKenna Burner**

To perform quantitative LII measurements, the instrument must be calibrated using a well-characterized sooting flame. Tomographically inverted light-extinction measurements were performed to determine soot volume fractions in the fuel-rich ethylene/air flat flame described previously. An example of a spatially resolved radial soot profile is shown in Fig. 8 for a height-above-burner of 25 mm. The radial profiles for each vertical position were averaged from R = 0-26 mm, as the soot distribution was found to be sufficiently flat. These averaged results are shown in Fig. 9. This radial average is treated as the vertical distribution of the horizontally uniform soot volume fraction in the premixed flat flame, which can then be used to calibrate the sensitivity of our LII instrument.

For LII-calibration measurements, it was found that the McKenna burner surface and stabilization plate obstructed signal collection from the calibration flame with the LII collection optics oriented at 39º to the facility.
floor. As a remedy, the laser sheet was rotated to a vertical alignment and the optical components removed from the cooling jacket and aligned on a horizontally-oriented optical rail to provide LII signal collection in an arrangement which was unobstructed by the McKenna burner setup. Rayleigh scattering (notch filter removed from LII setup) and LII images acquired from the McKenna burner are shown in Fig. 10. The square boundary of the fiber-imaging bundle is readily apparent in both images. The Rayleigh-scattering image was recorded to document the in-plane structure of the LII laser sheet within the field of view. The anomaly at the lower-right of the Rayleigh image results from scattered light from the burner surface, while the left half of the Rayleigh image reflects the in-plane distribution of light intensity within the laser sheet. The Rayleigh signal was plotted for a single column of pixels taken from the left-hand side of the image to better quantify the laser-sheet energy distribution, which is plotted to the left of the Rayleigh image in Fig. 10(a). The LII image data from the 1-cm square region at the center of the McKenna burner reflect an essentially flat soot distribution there, along with the expected rise in LII signal in the vertical direction resulting from a vertical soot distribution of the type shown in Fig. 9. Comparing the LII signal from images typical of Fig. 10(b) with the vertical soot profile of Fig. 9, we arrive at an initial estimate of 6000-10,000 counts/ppm of soot for the sensitivity of our LII instrument.

To minimize any potentially large measurement biases resulting from time-varying absorption of laser-sheet energy by in-fire soot in the path of the LII laser sheet, it is critical that the LII measurements be conducted in the “plateau-level” regime, where the LII signal is nearly independent of laser-pulse energy. The plateau-level response of the LII signal was checked by varying the laser-sheet energy delivered to the LII measurement volume within the McKenna burner flame. The LII signals were averaged over 100 laser shots and integrated LII intensities were extracted from images similar to the one shown in Fig. 10(b). The CCD chip was

![Figure 10. Rayleigh (a) and LII (b) images acquired of the calibration flame with the LII probe.](image1)

![Figure 11. Integrated LII intensity vs. laser sheet energy.](image2)
integrated in five horizontal areas of 21 × 201 pixels at different heights within the flame and corresponding to a nominally 0.5-mm × 5-mm × 0.3-mm volume. The plateau-level response of the LII system was then checked by plotting these integrated LII intensities vs. the pulse energy delivered by the laser sheet, as shown in Fig. 11. The results display a plateau-level response that is typical of LII generated from uniform-intensity laser sheets. The LII signal rises to a maximum, where, presumably, laser heating of the soot particles is sufficient for significant vaporization of soot to occur. The signal level then decays with increasing laser-pulse energy and becomes nearly constant, as signal loss resulting from soot vaporization is now balanced by LII signals which are generated within the lower energy wings of the laser sheet, where the fluence begins to reach threshold levels, or by non-LII processes such as Swan-band emission from laser-produced C2. Based on inspection of the curve shown in Fig. 11, the plateau-level region for this optical system is quite broad, and present for laser-sheet energies between ~60-200 mJ/pulse, so that a factor of 3 variation in laser-sheet energy will not significantly impact the measured LII signal in a pool-fire environment.

C. LII Measurements in Sooting Pool Fires

A photograph of a 30% toluene in methanol pool fire is provided in Fig. 12. Scattering of the 532-nm laser sheet is clearly visible at the mid-height of the fire and emission from hot soot can be seen throughout the fire plume (yellow-orange emission). Representative LII images from a 30%-toluene/70%-methanol pool fire are shown in Fig. 13. The false-color scale of these images represents the raw LII signal in detector counts, as we estimate that the uncertainty in our current calibration of the LII signal to be as high as 40%. To our knowledge, these are the first spatially resolved images of in-fire soot obtained within a fire plume of meaningful size. The LII signal is proportional to soot volume fraction and the images in Fig. 13 reveal the evolution of the fine-scale soot structure in a 1-cm square field of view located 1 m above the center of the liquid fuel pan. The projected size of an image pixel into the fire object plane is 25 µm square, so that resolution of submillimeter soot features in our LII images is readily obtained. In some cases, particle-like features can be seen in the LII images, which are present when LII is detected with both prompt and time-delayed gates, eliminating the possibility of intense laser light scattering as a cause for these particle-like interferences. The cause of this “contamination” of the LII signal is still uncertain at this point, but we believe the source to be either fluorescence or incandescence originating from remnants of a high-temperature polymer sealant used at the base of the fuel pan and entrained into the fire, which will be eliminated in future experiments. The mean detected LII signal level with the 100-ns prompt gate employed was ~5000 detector counts above background, which corresponds to a mean soot volume fraction of 0.5 to 0.83 ppm, based on the estimated 6000 to 10,000 count/ppm sensitivity of our LII instrument. These order 0.1-ppm values are reasonable for the methanol-diluted fuel blend used here.

Initial estimates of the shape of the soot-volume-fraction pdf at the center of the turbulent pool fire were obtained by constructing a histogram of the LII signal within a 350- × 350-pixel region and over an ensemble of 500 LII images of the type shown in Fig. 13. The resulting pdf of the LII signal is displayed in Fig. 14. The soot loading at the pool-fire center displays a “clipped” pdf, with the highest probabilities occurring at near-zero values and a long decaying tail that is representative of a relatively low mean signal with very high intermittency. Using the same ~6000 to 10,000 count/ppm sensitivity as above, the maximum detected soot-volume-fraction fluctuations are in the range of 6.5 to 10.9 ppm, as detected at the upper limit of our ICCD detector’s 16-bit dynamic range. At present, we are working to improve the approximately 40% uncertainty in the calibration of our LII instrument.

Figure 12. Digital photograph of a 30% toluene in methanol pool fire. Scattering from the LII laser sheet can be seen in the center of the image.
Figure 13. LII images acquired from a 30% toluene/methanol pool fire.

Figure 14. PDF of LII images acquired from 30% toluene/methanol pool fire.
IV. Summary and Conclusions

We have presented single-shot dual-pump CARS temperatures and LII images acquired independently from sooting toluene in methanol blended-fuel pool fires. The thermometry data indicate the capability of the CARS diagnostic to cope with appreciable levels of soot without succumbing to optical thickness effects, and to additional complications arising from C2 Swan and Raman-resonant emission. Additionally, it has been demonstrated that well-resolved LII images can be obtained from these large fires using our custom-fabricated, water-jacketed and fiber-coupled LII optical probe. A broad plateau-level region for the dependence of the LII signal on laser-sheet energy was verified, which is critical for quantitative LII measurements in turbulent fire plumes, where the soot absorption of laser energy delivered to the measurement volume will vary significantly on a shot-to-shot basis. The current uncertainty in the calibration of the LII instrument is estimated to be as high as 40%, which is attributed to uncertainty in the extinction volume-fraction measurements used to characterize the premixed calibration flame, and also potentially as a result of non-uniformities in the thickness of the LII laser sheet, to which plateau-level LII signals may be quite sensitive. Our present efforts are aimed at reduction of the uncertainty in the LII calibration and at combination of the CARS and LII systems for simultaneous data.

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