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Review of soot measurement in hydrocarbon-air flames

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Soot, which is produced in fuel-rich parts of flames as a result of incomplete combustion of hydrocarbons, is the No. 2 contributor to global warming after carbon dioxide. Developing soot measurement techniques is important to understand soot formation mechanism and control soot emission. The various soot measurement techniques, such as thermophoretic sampling particles diagnostics followed by electron microscopy analysis, thermocouple particle densitometry, light extinction, laser-induced incandescence, two-color method, and emission computed tomography, are reviewed in this paper. The measurement principle and application cases of these measurement methods are described in detail. The development trend of soot measurement is to realize the on-line measurement of multi-dimensional distributions of temperature, soot volume fraction, soot particle size and other parameters in hydrocarbon-air flames. Soot measurement techniques suitable for both small flames in laboratories and large-scale flames in industrial combustion devices should be developed. Besides, in some special situations, such as high-pressure, zero gravity and micro-gravity flames, soot measurement also should be provided.

hydrocarbon-air flames, combustion measurement, soot, laser-induced incandescence, emission CT

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1 Introduction

Hydrocarbon fuels are the main energy source in China. Soot is one of the most important pollutants emitting from hydrocarbon-air combustion. Soot formation and control directly relate to environment and humanity health [1–3]. When hydrocarbon fuels burn with inadequate air, soot precursors form and they will nucleate and grow into soot particles, which will decrease combustion efficiency. Soot staying in atmosphere may affect weather and climate, which also contributes to global warming [4]. In addition, soot particles will carry plenty of toxicants (such as heavy metal and organic matter) because of its high adsorption. These soot particles with diameters from 0.01 μm to 0.1 μm can be breathed into alveolus. Soot precursors, polycyclic aromatic hydrocarbons (PAHs), is a kind of carcinogen, which can harm breath system of human. Besides, soot particles will affect radiative heat transfer significantly. In combustion chambers, only a few soot particles will produce bright light, which will enhance radiative heat transfer of flames [5].

Practical combustion devices, including furnaces, gas turbines, and internal combustion engines, often emit soot. To decrease soot generation in these combustion devices, the processes of soot formation and burnout in different combustion processes should be understood. Detailed descriptions of chemical kinetics in diffusion hydrocarbon-air flames have used in soot formation models by many researchers, and various numerical simulations have been done [6–8]. However, because of variation of temperature and soot volume fraction in diffusion flames, the processes of soot formation and burnout, and chemical reaction are very complex. Developing soot measurement techniques is

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important to the understanding of physical and chemical mechanisms of soot formation and the validation of soot formation models. It is also helpful to increase combustion efficiency and decrease pollutant emission.

In this paper, soot measurement methods of hydrocarbon-air flames will be reviewed in detail. Then, the development trends of soot measurement are discussed. Hopefully, this review will provide researchers with an up-to-date knowledge about the applications of soot measurement in hydrocarbon-air flames.

2 Principles of soot measurement techniques

As shown in Figure 1, soot measurement techniques can be classified into two categories depending on the nature of contact, which exists between the measuring device and the flame.

In invasive measurement, the measuring device is in direct contact with the flame. For example, soot particles have often been collected by sampling techniques for subsequent analysis of soot microstructure, primary particle size and chemical composition by off-line apparatus such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and electron spectroscopy for chemical analysis (ESCA). Thermocouple particle densitometry (TPD) can be used to simultaneously measure flame temperature and soot volume fraction. However, invasive measurements are difficult to archive high temporal and spatial resolutions and they will disturb the measured flame. The most widely used soot measurement techniques are the noninvasive optical method.

Optical measurement techniques of soot in hydrocarbon-air flames are divided into two categories. One kind of measurement techniques are based on laser or other additional light source, which mainly includes light extinction

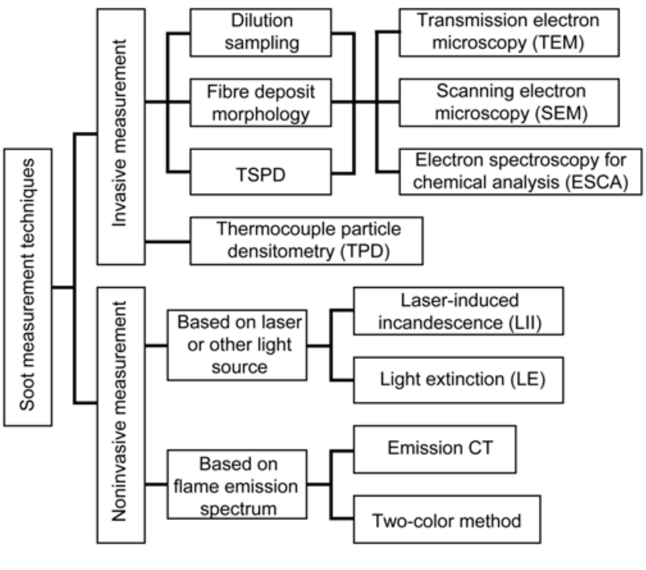

Figure 1 Soot measurement techniques.

(LE) and laser-induced incandescence (LII). Optical measurement techniques based on laser have become a most popular diagnostic method on combustion. However, laser diagnostic measurement techniques make use of a large number of sophisticated instruments. Another kind of measurement techniques are based on flame emission spectrum. It directly detects integral values of soot emission and then calculates temperature and soot volume fraction from the flame emission spectrum. Typical emission techniques include two-color method and emission computed tomography (emission CT).

The principles of these soot measurement techniques will be described as below.

2.1 Thermophoretic sampling particle diagnostics followed by electron microscopy analysis

In soot sampling techniques shown in Figure 1, dilution sampling can measure the quantities and sizes of soot particles, and fibre deposit morphology can disclose the spatial distributions of soot particles in flames. However, both the two methods cannot obtain instantaneous soot microstructure in any spatial position of flames. Thermophoretic sampling particle diagnostics (TSPD) adopts thermophoresis for collecting soot particle/aggregate in flames and the microstructure and size of the sampled soot will be observed and analyzed by SEM or TEM [9, 10]. The principle of TSPD is briefly described as below. Small particles will move from the high temperature region to the low temperature region because of the temperature gradient in flames, which is also called thermophoretic mass transfer. When a probe is inserted into flames, thermophoretic deposition is driven by the presence of a temperature gradient in the surface of the cold probe inside the flow field of a particle-laden hot gas. The probe exposure time should be long enough to capture a significant soot sample but short enough to present a cold surface to the flame-born particles. This cold surface serves a second important purpose, which is that it freezes heterogeneous reactions of the particles that are already captured. This chemical freezing action prevents changes in the soot morphology after the particles have impacted on the cold surface.

A typical TSPD system is shown in Figure 2 [11]. A double-acting probe is operated to insert into the flame for dozens of microseconds. A copper TEM grid is installed at the tip of the probe. When the probe is inside the flame, soot particles are thermophoretically deposited onto the grid. The soot samples collected on the gird are subsequently examined by electron microscopy. Because TSPD is an invasive measurement technique, the probe enters the flame very rapidly, which will disturb the flame. To decrease the disturbance to the flame, the thickness of the probe is very thin.

Because the sizes of soot particles are very small, the microstructure of soot can only be observed by SEM or TEM. Figure 3 shows a TEM graph of soot particles sampled in a diffusion ethylene-air flame. From Figure 3, it can be seen that primary soot particles are generally small and spherical, ranging in size of dozens nanometers. The composition of soot particles are carbon, and less hydro and oxygen through analysis of ESCA.

2.2 Thermocouple particle densitometry

McEnally et al. [12] successfully measured soot volume fraction in flames through analysis of variation of measurement results in a sampling probe (such as thermocouple) caused by soot deposits. Their research showed that soot deposits in a thermocouple is based on thermophoretic theory, in which the particle deposition rate to a cold surface immersed in a flame is dominated by thermophoresis, instead of Brownian diffusion. The thermophoretic mass flux of particles to the junction surface can be written as

$$j_T = (D_T Nu_j f_v \rho_p / 2d_j)[1-(T_j / T_g)^2], \quad (1)$$

where j_T is the mass flux to the junction per unit time and surface area; D_T stands the soot particle thermophoretic diffusivity; N_u stands for the Nusselt number for heat transfer; f_v is the soot volume fraction; ρ_p is the density of soot particle; d_j is the diameter of junction of the thermocouple; T_j is the junction temperature; T_g is the flame temperature.

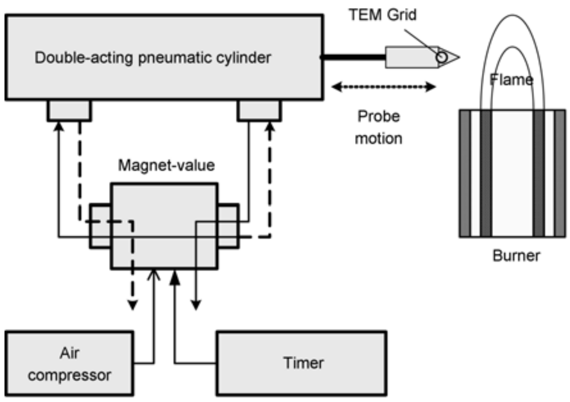

Figure 2 Schematic diagram of thermophoretic sampling particle diagnostics (TSPD) system [11].

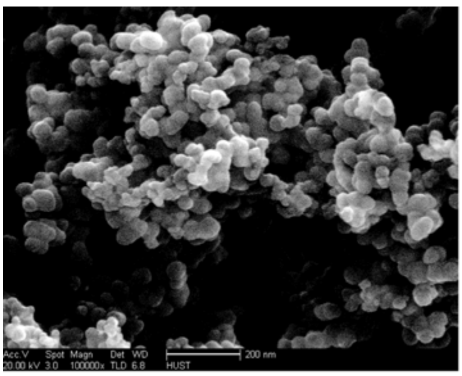

Figure 3 TEM graph of soot particles.

If heat generation by radiation from the junction to flame and heat transfer by convection between the junction and flame are considered, then a quasi-steady energy balance at the junction is as follows.

$$\varepsilon_j \sigma T_j^4 = (k_{g0} Nu_j / 2d_j)(T_g^2 - T_j^2), \quad (2)$$

where ε is the emissivity of the junction of the thermocouple; σ stands the Stefan-Boltzmann constant; k_{g0} stands for the ratio of conduction coefficient of air and flame temperature.

The measurement process of TPD is described as below. The thermocouple is inserted into a sooting flame rapidly. Soot particles will deposit on the junction of the thermocouple. The variation of the junction temperature contains three separate stages, as shown in Figure 4, which are transient-response stage, variable-emissivity stage, and variable-diameter stage. The junction temperatures of the thermocouple in three stages are recorded and then the temperature and soot volume fraction will be measured according to eqs. (1) and (2).

There are some advantages of TPD. Firstly, it directly obtains spatial distributions of local temperature and soot volume fraction in flames. Secondly, TPD normally will not require the construction of any complex apparatus and reconstruction methods, and therefore has a very low set-up cost. TPD does not depend on the refractive index function of soot particles. Thus, TPD could be useful for calibration of noninvasive methods that only provide relative soot volume fractions. However, it can not obtain spatially and temporally resolved results for unstable flames. Moreover, it will disturb the measured flame.

2.3 Light extinction

Light extinction is a path-integrated or line-of-sight technique. When a beam of light passes the flame, the light will be attenuated by soot particles. The attenuation can be de-

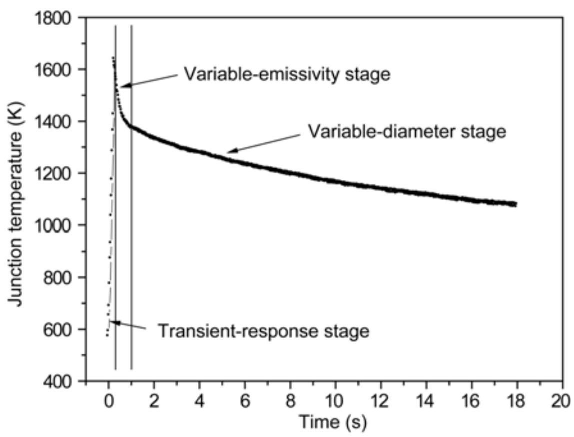

Figure 4 The variation of the junction temperature with time measured in an ethylene-air diffusion flame.

scribed by Beer-Lambert law as below:

$$\tau = I / I_0 = \exp\left(-\int_0^L \kappa_\lambda \mathrm{d}l\right), \quad (3)$$

where τ is the transmission; I is the intensity of transmission light; I_0 is the intensity of incidence light; L is the length passed by the light; κ_λ the absorption coefficient.

For particles in the Rayleight approximation, the soot volume fraction is given by

$$f_v = \kappa_\lambda \lambda / \left[6\pi E(m)\right] = \ln(\tau)\lambda / \left[6\pi L E(m)\right], \quad (4)$$

where $E(m)$ is the refractive index function of soot; λ is the wavelength of the light.

The technique provides field-integrated values of the soot volume fractions. Full-field light extinction has been used to measure field distributions of soot volume fractions in uniform flames. For a non-uniform flame, eq. (4) can be written in its differential form as [13]

$$f_v(r) = \frac{\mathrm{d}\ln(\tau)}{\mathrm{d}r} \lambda / \left[6\pi L E(m)\right]. \quad (5)$$

Therefore, an inversion method is needed to reconstruct the spatial soot volume fraction distribution in flames, e.g. the Abel inversion method for axisymmetric flames [13, 14].

The measurement system of light extinction needs an additional light source, such as laser or arc lamp. A schematic diagram of the experimental system of a full-field light extinction is presented in Figure 5 [14]. The laser beam passes through a neutral density filter, a beam expander, a set of diffusers, and a collimator before it encounters the soot-laden flame. Then, the light passes through a decollimating lens, bandpass and neutral density filters. Finally, the light is received by a CCD camera. Each pixel associated with a corresponding digital image records the intensity I'. When the flame is extinguished, the intensity is I'_0. Firstly, an adjustment must be made for the flame radiation by obtaining another image of the flame (with the corresponding pixel intensity I_f) with the laser switched off. The second correction for ambient light requires that an image should be obtained in the absence of both the flame and the collimated laser beam. If the corresponding pixel intensity of ambient light is I_b, the transmittance is calculated as below:

$$\tau = I / I_0 = (I' - I_f) / (I'_0 - I_b). \quad (6)$$

Some researchers [11, 15] used a spectroscope to obtain a reference light, and then used two photodiodes to receive the transmitted light and reference light. The light signals passing flame before and after were simultaneously detected and transferred into a computer to be processed. The whole measurement procedure was simplified.

2.4 Laser-induced incandescence

Laser-induced incandescence has become a popular technique for measurements of soot volume fraction. LII involves heating particles with a high power pulsed laser and analyzing the thermal radiation from the hot particles, and it is used to obtain the two-dimensional (2-D) distributions of soot volume fraction. The principle of LII is briefly described as below [16, 17]. With a planar laser with high fluence to heat soot particles in flame, when temperature of soot is high enough (over 4000 K), it will emit incandescence. The LII signal is proportional to soot volume fraction, as shown in eq. (7). The temperature of soot particles will decrease to the flame temperature after several hundred ns, and the LII signal disappears. An ICCD with bandpass filters is used to receive the LII signal. A reference object with known soot volume fraction is taken for the absolute quantification (calibration) of the LII signal. After calibration, the LII signal can be used to obtain 2-D distributions of soot volume fraction. Figure 6 shows a schematic diagram of experimental set-up for measurement of a hydrocarbon-air flame using LII.

$$S_{LII} \propto \frac{8\pi c^2 h E(m)}{\lambda^6} d_p^3 \exp\left(-\frac{hc}{\lambda k T}\right), \quad (7)$$

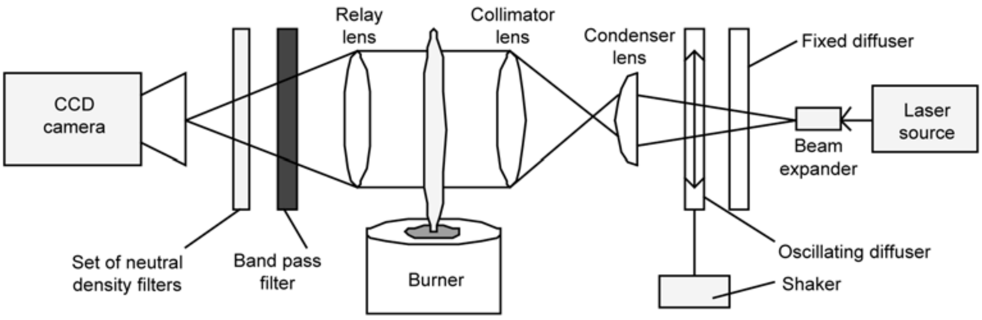

Figure 5 Schematic diagram of the experimental setup of full-field light extinction [14].

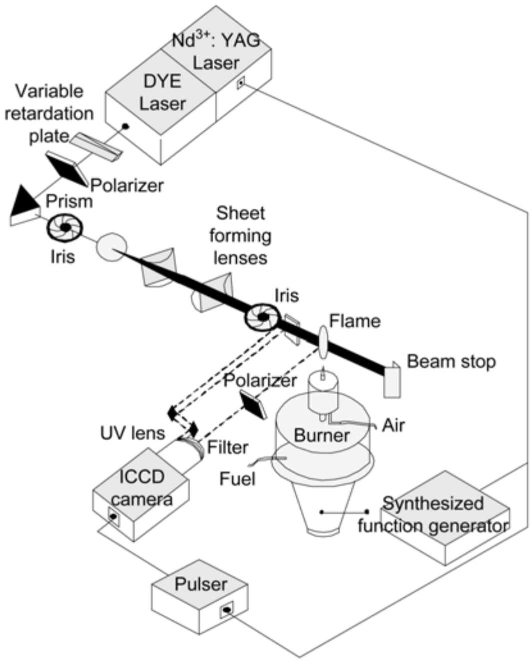

Figure 6 Schematic diagram of the experimental setup of laser-induced incandescence system [18].

where c is the speed of light; h is the Planck constant; d_p is the diameter of soot particle; k is the Boltzmann constant.

A two-color LII technique for quantitative soot volume fraction measurement by detection of the absolute LII intensity has been proposed by Smallwood et al. [19]. Through a proper calibration, the absolute LII intensity at two wavelengths can be obtained by two ICCD cameras. The temperature of soot particle can be determined from their ratio by two-color method, and then the soot volume fraction can be calculated as follows.

$$T_p = \frac{hc}{k}\left(\frac{1}{\lambda_2}-\frac{1}{\lambda_1}\right)\left[\ln\left(\frac{V_{\exp_1}\lambda_1^6}{\eta_1 E(m_{\lambda_1})}\right)-\ln\left(\frac{V_{\exp_2}\lambda_2^6}{\eta_2 E(m_{\lambda_2})}\right)\right] \quad (8)$$

where $V_{\exp1}$ and $V_{\exp2}$ are the intensities of LII signal in two gains, respectively; η is the correct factor of the system.

$$f_v = \frac{V_{\exp}}{\eta w_b G_{\exp}\dfrac{12\pi c^2 h}{\lambda^6}E(m_\lambda)\left[\exp\left(\dfrac{hc}{k\lambda T_p}\right)-1\right]^{-1}}, \quad (9)$$

where $G_{\exp}$ is the gain of the system; ω_b is the thickness of planar laser.

LII not only provides information on soot volume fraction, but also can be used to determine soot particle diameter information according to the decay time of LII signal. However, LII models, which incorporate complex and competing mechanisms, typically account for particle heating by laser absorption and cooling by conduction to surrounding gases, sublimation of carbon clusters, and emission of thermal radiation. Different models will give different size distributions of soot particles [20]. More efforts will be put into the development of LII models. Besides, soot particle heating is significantly influenced by some experimental parameters, such as the excitation wavelength and laser fluence. The wavelength and power of laser should be determined carefully [20, 21].

2.5 Two-color method

The two-color method is a most widely used emission spectrum technique [22]. The flame temperature can be calculated with the monochromatic radiative intensities in two different wavelengths as

$$\left[1-\left(\frac{e^{(C_2/\lambda_1 T)}-1}{e^{(C_2/\lambda_1 T_{a1})}-1}\right)\right]^{\lambda_1^{\alpha_1}} = \left[1-\left(\frac{e^{(C_2/\lambda_2 T)}-1}{e^{(C_2/\lambda_2 T_{a2})}-1}\right)\right]^{\lambda_2^{\alpha_2}}, \quad (10)$$

where T is the flame temperature; T_{a1} and T_{a2} are the apparent temperatures in two wavelengths; C_2 is the second Planck's constant; the value of the parameter α depends on the physical and optical properties of the soot in the flame. In practice, flame monochromatic emissivity can be estimated using radiation law with known flame temperature. Then, the KL factor of the flame will be calculated using Hottel and Broughton equation from flame monochromatic emissivity as follows

$$KL = -\lambda^{\alpha}\ln\left[1-\left(\frac{e^{C_2/\lambda T}-1}{e^{C_2/\lambda T_a}-1}\right)\right], \quad (11)$$

where K is the absorption coefficient; L is the geometric thickness of the flame along the optical axis of the detection system. It has been proven that KL factor is proportional to the soot volume fraction in the flame.

The two-color method will only give a true flame temperature if a spatially uniform temperature exists along the line of sight. However, the non-uniformity of temperature and soot volume fraction will affect the physical meaning of the measured results [22]. The non-uniformity of soot volume fraction has less effect on the measured flame temperature than dose the non-uniform temperature on the KL factor. If a severe non-uniform distribution of temperature exists, the KL factor will be underestimated while the temperature measured by the two-color method is higher than the true temperature.

2.6 Emission computed tomography

For a non-uniform flame, emission CT technique based on inverse calculation can be used to simultaneously obtain distributions of temperature and soot volume fraction from emission spectrum of the flame. It is also a kind of measurement technique based on line-of-sight. Generally, a spectrometer or CCD image sensor is used to scan a cross section of flame, in order to obtain spectrum radiation information from different directions. In visible or infrared spectrum, the received radiation is mainly come from soot particles. As shown in Figure 7, monochromatic radiative intensity from the *j*th line-of-sight of an axisymmetric flame can be expressed as [23, 24]

$$I_\lambda(j)=\int_{l_0(j)}^{l_1(j)}\kappa_\lambda(l)I_{b\lambda}(l)\exp\left[-\int_{l}^{l_1(j)}\kappa_\lambda(l')\mathrm{d}l'\right]\mathrm{d}l, \quad (12)$$

where $I_{b\lambda}$ is the monochromatic radiative intensity of blackbody.

Actually, eq. (12) is the radiative heat transfer equation with non-scattering medium and cold blackbody boundaries. Simultaneous reconstruction radiation source (temperature) and radiative properties of medium (soot volume fraction) from this equation needs to solve the inverse radiative problems. For asymmetric flames, the similar calculation model can also be established according to radiative heat transfer equation. However, because of the complexity of the solution of the inverse radiative problems, the flame is assumed to be optically thin and self-absorption of soot particle is neglected in many researches, which can decrease the complexity of inverse radiation problem. An inversion scheme (e.g. Abel inversion method) is used to reconstruct temperature and soot volume fraction from multi-wavelength radiative emission data of flames [23]. However, self-absorption of soot will be considered in large-scale flames in industrial combustion chambers.

3 Application of soot measurement

Developing soot measurement techniques is useful to understand soot formation mechanism. It is also helpful to increase combustion efficiency and decrease pollutant emission. Soot measurements generally include measurement of soot microstructure, charged characteristic, primary particle size, distributions of soot volume fraction and flame temperature, which can be used to analyze soot particles inception, formation, aggregation, and oxidation processes.

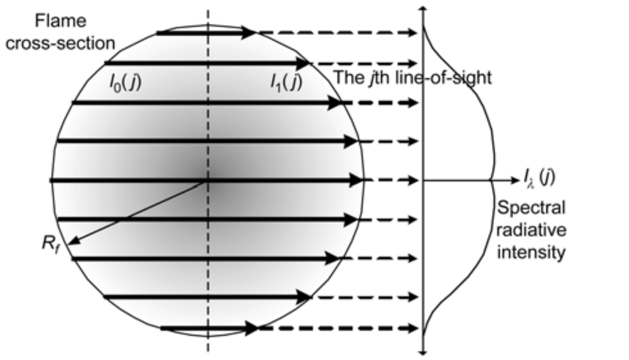

Figure 7 1-D tomographic reconstruction from line-of-sight spectral radiative intensity of flame emission.

The application of measurement techniques including TSPD-TEM, TPD, LE, LII, two-color method, and emission CT will be summarized as below.

3.1 Thermophoretic sampling particle diagnostics combined with electron microscopy

Generally, sampling techniques combined with off-line electron microscopy analysis are used to analyze soot microstructure and charged characteristic. TSPD followed by TEM analysis is most widely used. The size of soot primary particle is about 20 nm. With surface growth and oxidation of soot, these primary particles form soot aggregates. The primary soot particles include structural mixtures and closed-shell structure observed by TSPD-TEM. The former is divided into liquid-like carbon and amorphous carbon while these closed-shell structures are characterized by onion-shell structure and carbon nanotubes [11]. TSPD-TEM can provide complement information for optical measurements. In addition, TSPD-TEM could also be useful for calibrating methods such as LII [25].

3.2 Thermocouple particle densitometry

TPD is mainly used in measurement of temperature and soot volume fraction in laminar diffusion flames in laboratories [12, 26, 27]. Although the probe contacts with the flame in the measurement, this method obtains information through the variation of temperature in the probe because of soot deposition. It is different from other invasive measurement techniques, in which the sampled soot particles will be analyzed by other apparatus. TPD is very simple to implement experimentally and can simultaneously measure temperature and soot volume fraction. In ref. [24], a thermocouple employed in measurements of an axisymmetric laminar diffusion flame generated by a Gülder burner was Pt-Pt/10%Rh wire pairs, as shown in Figure 8. The diameter of the junction was 240 μm and the wire diameter was 75 μm. The distributions of flame temperature and soot volume fraction measured are shown in Figure 9. It can be seen that TPD can obtain soot volume fraction in the under part of the flame near the outlet of the burner, in which PAHs are gen-

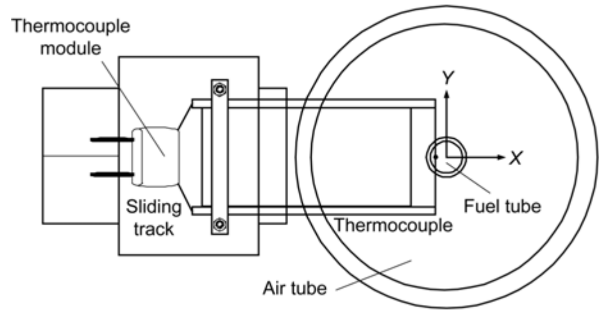

Figure 8 Diagram of the thermocouple arrangement for co-flowing non-premixed burner. The view is from the above [12].

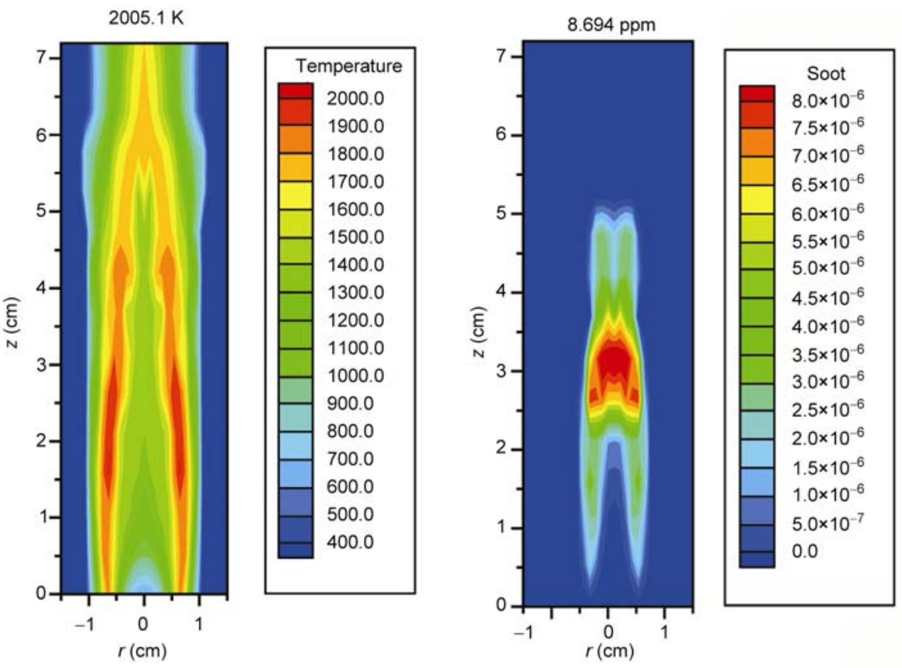

Figure 9 Temperature and soot volume fraction of ethylene-air diffusion flame measured by TPD.

erated. In the upper part of the flame, the concentration of oxygenation medium is sufficient to rapidly oxidize any soot particles, which will take some uncertainties for measurement of soot volume fraction.

However, as a contacting measurement technique, TPD is limited in application. Recent years, researchers have proposed that the radiation from soot particles to the junction and the heat conduction between the junction and the wire should be considered in the quasi-steady energy balance of the junction of thermocouple [27, 28], which will improve the accuracy of TPD.

3.3 Light extinction

Light extinction is a commonly used diagnostic technique for measuring soot volume fraction from 1970s [13–15, 29, 30]. It can give a true soot volume fraction in a uniform flame, e.g. a flat flame generated by a McKenna burner [11].

For a non-uniform flame with characteristic distribution, e.g. an axisymmetric laminar diffusion/premix flame generated by a Santoro burner or a Gülder burner, the soot volume fraction can be obtained by an inversion scheme such as Abel inversion method. Snelling et al. [13] demonstrated a technique for acquiring 2-D soot volume fraction in a laminar flame with an arc lamp source. Two dimensional attenuation measurements at 577 nm were presented and Abel inverse method was used to obtain radial profiles of soot concentration in an axisymmetric laminar diffusion ethylene-air flame. Arana et al. [14] reported that a full-field light extinction measurement was used to measure soot volume fraction distributions in partially premixed laminar coflow ethylene flames. The effects of equivalence ratio on soot generation were analyzed. Thomoson et al. [15] measured soot volume fractions in a laminar nonpremixed methane-air flame over the pressure range of 0.5 to 4 MPa. It has been observed that the maximum soot concentration depended on pressure according to a power law and peak carbon conversion to soot also followed a power-law dependence on pressure. Daun et al. [31, 32] proposed a method based on Tikhonov regularization instead of Abel inversion for reconstruction of soot volume fraction in an axisymmetric flame in measurement of light extinction. The refractive index function of soot $E(m)$ should be given in advance in light extinction, but how to select $E(m)$ is a challenge to researchers.

Light extinction provides field-integrated values of the soot volume fractions for a non-uniform flame without characteristic distribution. In ref. [33], the temperature of carbon dioxide in the alcohol flame and the soot temperature in the candle flame were determined using FTIR emission-transmission spectra. In ref. [34], a study was performed to obtain the average volume concentration of soot particles in a butane flame by the extinction method. Combined with the refractive index of soot particles in visible and near infrared wavelength ranging from 514.6 nm to 1063.3 nm, the flame monochromatic emmissivities versus wavelength and combustion temperature were derived.

As mentioned in section 2.3, flame emission will affect measurement of light extinction. However, combining measurement of light extinction and flame emission, distributions of soot volume fraction and temperature in an axisymmetric flame can be simultaneously obtained [35]. For the axisymmetric flame, the outgoing spectral transmission radiative intensity of a beam of laser can be expressed as

$$I_{1,\lambda}(j)=\int\kappa_{\lambda}(l)I_{b\lambda}(l)\exp[-\int\kappa_{\lambda}(l')\mathrm{d}l']\mathrm{d}l + I_{0,\lambda}\exp[-\int\kappa_{\lambda}(l')\mathrm{d}l'], \quad (13)$$

where $I_{0,\lambda}$ is the monochromatic radiative intensity of the laser.

When the laser is turned off, the outgoing emission radiative intensity $I_{2,\lambda}$ of the flame in the line of sight can be expressed as eq. (12). The transmittance of flame is

$$\tau=(I_{1,\lambda}-I_{2,\lambda})/I_{0,\lambda}. \quad (14)$$

Firstly, the distribution of soot volume fraction is retrieved from the transmittance by Abel inversion method, and then based on the previous retrieval of soot volume fraction, the temperature distribution is reconstructed from the emission radiation of flame.

In ref. [36], a multi-wavelength inversion method was extended to reconstruct the time-averaged temperature distribution in a non-axisymmetric turbulent unconfined sooting flame by the multi-wavelength measured data of low time-resolution outgoing emission and transmission radiation intensities.

In a word, light extinction is mainly used in of measurements of laboratory scale axisymmetric laminar diffusion flames. If this technique is used to measure non-uniform flames, the layout of light and the reconstruction method should be improved. Moreover, light extinction is used to calibrate laser-induced incandescence.

3.4 Laser-induced incandescence

In 1977, Eckbreth [37] observed a disturbing light, LII signal, emitted by soot particles, which were heated by laser, in flames in Raman scatter experiments. In 1984, Melton [16] and Dasch [38] established theoretic models of measurement of soot volume fraction and primary particle size. In the models, the proportional relationship between LII signal and soot volume fraction was obtained. In the following twenty years, LII became a popular technique for measurements of soot concentration and primary particle size because it is a noninvasive, in-situ measurement technique and with good sensitivity, high spatial and temporal resolution [16–27, 37–42].

At present, the researches of soot measurement by LII include two categories. In one category, the implementation of LII are studied by relying on the improvement of calibration, LII models, excitation and detection wavelengths, laser fluence and determination of the refractive index function. Vander Wal et al. [39] indicated that LII signal could be separated from other disturbing light such as fluorescent or scattering light through adjusting some parameters, e.g. laser wavelength, detection wavelength, the speed of shutter, and delaying time. It will get more accurate results. In another category, the characteristic of soot particles and aggregates induced by laser irradiation were studied. Smallwood et al. [19] analyzed the heat transfer of soot aggregates detailedly and proposed the two-color LII for the determination of soot volume fraction. In 2005, an international workshop of LII was held in Duisburg, Germany. The workshop focused on fundamental experimental and theoretical aspects of soot measurement by LII. The goal of the workshop was to review the current understanding of the technique and identify gaps in this understanding associated with experimental implementation, model descriptions, and signal interpretation [20]. It was decided that the international workshop of LII would be hold every two years. Schluz et al. [20] reviewed recent trends and current questions of LII. Firstly, the modeling of LII signal involves particle heating by laser absorption and cooling by radiation, sublimation, conduction to the surrounding atmosphere. Moreover, it may involve other processes such as soot particle evaporation, oxidation, photodesorption, etc. Schluz et al. compared nine LII models which were frequently used, and the results of this comparison indicated that different models would give different results. Secondly, the effects on the measured results of many experimental parameters, e.g. excitation and detection wavelengths, laser fluence, refractive index function of soot, spatial beam profile, and temporal detection issues, were analyzed. Then, the relationships between soot volume fraction, soot primary particle size and LII signal were evaluated. Finally, the soot volume fractions and primary particle sizes measured by LII in McKenna burner, Gülder burner and Santoro burner were given.

In China, Wang et al. [21] carried out simulation investigation of LII based on the melton model. Wang et al. [11–41] set up an LII diagnostics system, with which the soot distributions and aggregates in electric field were studied. He et al. [42] investigated soot formation of bio-diesel fuel in the cylinder of IC engines. With the popular application of LII, the first workshop of LII in China was hold in Hefei in September 2009. In the workshop, the researchers of National Research Council of Canada introduced LII models and application of LII abroad and Tshinghua University presented soot measurement in flames in laboratory and diesel engine using LII.

3.5 Two-color method

Since the two-color method utilizes the thermal radiation from solid particles in combustion and directly measures their temperature without complex optical system, it is widely used in combustion diagnosis in the cylinder of internal combustion (IC) engines. Matsui et al. [43] firstly proposed that the luminous radiation of diesel flame comes from soot particles in diesel engine and the soot particles can faithfully follow the surrounding gas temperature. The combustion temperature and emissivity in the cylinder were obtained by processing thermal radiation of soot particles. Then the *KL* factor could be calculated from the emissivity. Yan et al. [44] measured the flame temperature and *KL* factor along the direction of the fiber in the cylinder of a Cummins NH engine. Chen et al. [45] analyzed the effects of multi-parameters on local instantaneous flame temperature and soot concentration in the cylinder of diesel engines using an improved two-color method. Tian et al. [46] studied the distributions of flame temperature and soot volume fraction in IC engine with two-color method. He et al. [47] analyzed the effect of biodiesel on the combustion and soot formation in the cylinder from measurement results by the two-color method.

From eqs. (10) and (11) it can be seen that the estimation of the *KL* factor depends on the values chosen for the parameter α and the wavelength. The value of α is a function of the soot particle size and the refractive index of soot. Fuel type may also have an influence on the value of choice of a suitable value for α [22]. In theory, it can choose any wavelength in the two-color method. However, the choice of wavelengths for the two-color method involves a number of considerations that must be taken into account. Firstly, the value of α depends on the light wavelength. Secondly, the spectral response of measurement system should be considered. At last, the emission spectrum of flame should be considered. Besides, in ref. [48], a sensitivity analysis of the two-color pyrometry technique for calculation of soot temperature and volume fraction was presented. The calculations were carried out by taking into account the particle shape, optical property variations and size distribution of soot particles.

To implement two-color measurement, the monochromatic radiation in two different wavelengths need to be obtained by beam splitters, filters and monochromatic CCD cameras. Recently, color CCD cameras have been used to capture monochromatic images in visible spectrum. In ref. [49], various imaging systems for separation of measurement wavelength bands used in two-color method were presented.

In eqs. (10) and (11), the two-color method is implemented though the apparent temperature. Besides, the two-color method can also be realized by ignoring the difference of emissivities in the two close wavelengths, as shown in eqs. (15) and (16). Yan et al. [50] concurrently measured temperature and soot volume fraction of flames in a single burner furnace using this kind of two-color method.

$$T = C_2\left(\frac{1}{\lambda_2}-\frac{1}{\lambda_1}\right)\ln\left(\frac{I_{\lambda 1}}{I_{\lambda 2}}\cdot\frac{\lambda_1^5}{\lambda_2^5}\right), \tag{15}$$

where $I_{\lambda 1}$ and $I_{\lambda 2}$ are the monochromatic radiative intensities in the two wavelengths.

$$I_{\lambda_1} / I_{b,\lambda_1}(T) = \varepsilon_{\lambda_1} = 1-\exp(-\kappa_{\lambda_1} L / \lambda^{\alpha}), \tag{16}$$

where $\varepsilon_{\lambda 1}$ is the emissivity of the flame.

Since the results measured by two-color method are the integrated value along the line of sight, the effect of spatial gradients of soot properties within the measurement volume were analyzed [51].

3.6 Emission computed tomography

Emission CT is a noninvasive measurement technique and is easy to implement without complex optical apparatus. It can simultaneously obtain non-uniform temperature and soot volume fraction by solution of inverse problem. Especially, in some special cases such as zero gravity or micro-gravity experiments [52] and large-scale combustion chambers, optical diagnostics based on laser are not suitable while emission CT is rather simple to implement. Many researches have carried out experiments for a long time [23, 24, 52–62].

De Iuliis et al. [53] compared the measured soot volume fractions of an axisymmetric ethylene-air flame by emission CT and light extinction. Good agreement was found between the two techniques. Snelling et al. [23] reconstructed temperature and soot volume fraction in axisymmetric laminar diffusion flames from multi-wavelength flame emission using Abel inversion method, when the attenuation of emitted flame radiation was ignored. The effects of the refractive index function of soot on measured results were discussed. Ayranci et al. [54, 55] proposed an inversion scheme for optically thin axisymmetric flames for in situ characterization of soot temperature and volume fraction fields via one-dimensional (1-D) tomographic reconstruction of line-of-sight flame emission spectra and the experiment was carried out within the 1.1 to 1.7 μm spectral range. Li et al. [56] used a CCD camera with filter to obtain monochromatic radiative intensity images in four wavelengths, from which the flame temperature and soot volume fraction in an axisymmetric flame were reconstructed. Thomson et al. [15] studied soot production in laminar non-premixed methane-air flames over the pressure range of 0.5 to 4 MPa using spectral soot emission measurement. Weikl et al. [52] used CARS and emission CT to simultaneously measure temperature distribution of an axisymmetric hydrocarbon-air flame in a parabolic flight experiment. The experimental results proved that emission CT is an appropriate and robust alternative for measuring temperatures in sooting axisymmetric flames, if more accurate techniques cannot be applied, e.g., during investigations in zero-g parabolic flight experiments.

With the development of emission CT, the studied object is extended to asymmetric flame from axisymmetric flame. In ref. [57], a high-resolution camera equipped with a stereo adapter was employed to capture stereoscopic flame images and a matrix-decomposition-based least squares algorithm was introduced to reconstruct flame temperature and soot volume fraction in an asymmetric flame. Huang et al. [58] used four CCD cameras to acquire flame radiation from different directions, from which flame temperature and soot volume fraction in an asymmetric flame were reconstructed. And the anti-noise capability of the radiative model has been assessed.

In previous researches, the flame was assumed as optically thin and self-absorption was neglected, as shown in the following equation:

$$I_{1,\lambda}(j) = \int \kappa_\lambda(l) I_{b\lambda}(l) \mathrm{d}l. \tag{17}$$

Snelling et al. [23] proved that the influence of the attenuation error was very small for a laboratory grade flame e.g. a flame generated by a Santoro burner or a Gülder burner. However, self-absorption should be considered in flames with higher soot loading in industrial applications.

Ai et al. [24, 59] detected flame radiative intensities in wavelengths of red, green, and blue, and then proposed an emission method, in which a simultaneous estimating procedure was carried out from the spectral radiative intensities by using a Newton-type iteration algorithm and the least-squares method. Liu et al. [60] used the conjugate gradient method and the 1-D search method to estimate the temperature profile and the absorption coefficient in an absorbing, emitting, non-scattering, and gray semitransparent parallel plane. In refs. [61, 62], a decoupled reconstruction method, which combines regularization methodology and optimization methodology, was proposed to simultaneously reconstruct flame temperature and soot volume fraction. Simulation research proved that the decoupled reconstruction method is of good accuracy and robustness. Experimental investigations have been done in an axisymmetric diffusion ethylene-air flame using a CCD camera with filter. The measured results are shown in Figure 10, where it can be seen that the soot volume fraction in the flame increases with the increase of the fuel flow rate. In addition, as the fuel mole fraction increases, the location of peak soot shifts from the center of the flame to the edge of the flame. Besides, the experimental results indicated that much more soot lies inside the flame zone with higher temperatures.

4 Development trend of soot measurement

The development trend of combustion measurement is from invasive measurement to non-invasive measurement, from off-line measurement to online measurement, from 1-D measurement to 2-D/3-D measurement, from measurement of one type of parameter to measurements of multi types of parameters. Moreover, suitable combustion measurement techniques should be developed for both scientific researches and industrial field. According to the application of those soot measurement techniques above, the development trend of soot measurement is discussed as below.

Contacting measurement techniques such as TSPD-TEM, TPD, etc. have obvious disadvantages, such as single-point measurement and invasive nature. These techniques are difficult to obtain spatially and temporally resolved results for unstable flames. However, since the measured results by these techniques do not rely on the refractive index function of soot particles, these techniques can be used as supplement diagnostics tools as optical techniques.

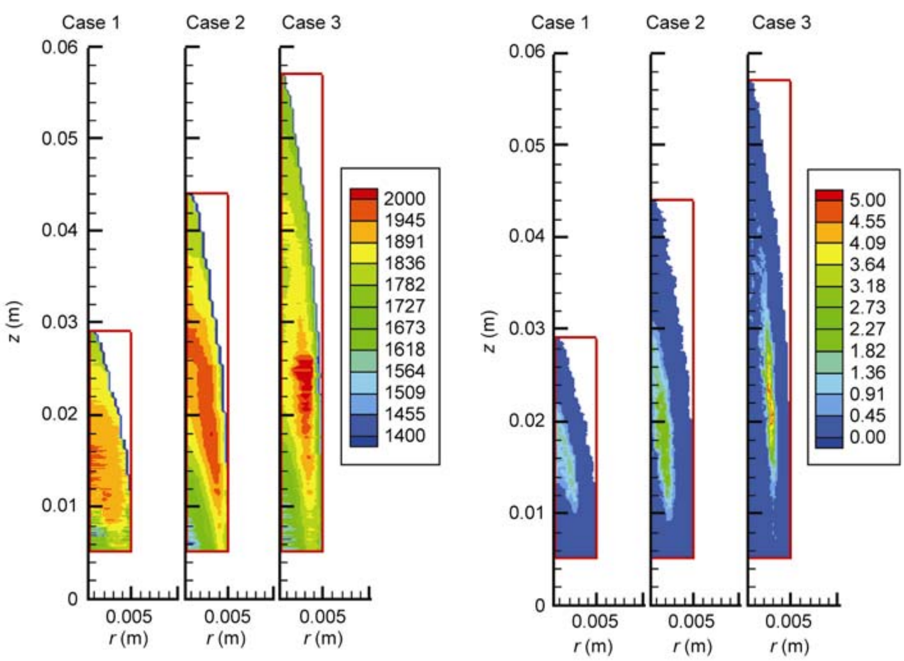

Figure 10 Temperature (Left) and soot (Right) volume fraction of ethylene-air diffusion flame measured by emission CT.

In optical techniques, both LE and LII will use laser or other additional light sources and the layout of optical system is complex. Besides that, an inversion method is necessary for reconstruction of the spatial soot volume fraction distribution in non-uniform flames. Moreover, only combined with emission measurement of flames, LE can simultaneously obtain distributions of soot volume fraction and temperature in axisymmetric flames. Therefore, the application of LE will be replaced by LII or emission measurement. However, light extinction can be used to calibrate laser-induced incandescence.

Although the two-color method has widely used in measurements in the cylinders of IC engines, it only obtains integrated values along line-of-sight. Two-color measurement can also be implemented through processing flame emission data when in emission CT measurement.

In conclusion, LII and emission CT will be most widely applied in future because of their advantages as follows. Both the two techniques are invasive measurement. LII can on-line measure 2-D distributions of soot volume fraction and size in flames. It will give highly accurate results after calibration. However, LII needs laser and other complex optical system. The conventional LII requires a calibration source whose soot volume fraction is known and uniform. On the other hand, laser fluence, LII signal models, and wavelengths will affect measurement of soot particle size. One of the characteristics of emission CT is simultaneous determination of 1-D/2-D distributions of temperature and soot volume fraction. The accuracy of emission CT has been validated. It is easy to implement and can be used in some special cases in which optical techniques based on laser are not suitable. Nevertheless, the radiation model of emission needs to be improved and more robust reconstruction algorithms should be developed. The challenges of the two techniques in future application will be discussed in the followings.

4.1 Laser-induced incandescence

LII is used for simultaneous measurements of soot volume fraction and size in flames. There are still some issues to deal with.

1) LII models have already considered particle heating by laser absorption and cooling by conduction to surrounding gases, sublimation of carbon clusters, and emission of thermal radiation. In a high temperature situation, how important are other physical and chemical processes such as annealing, photodesorption, soot oxidation, especially in nano-scale? Fundamental research is required to improve current understanding of LII models.

2) It has been proven that the refractive index function and thermal accommodation coefficient will affect LII models [63], but how can we determine these parameters in application? These LII models need to be compared with in a wide range of detailed experimental data.

3) There are some drawbacks of the current sublimation model. If laser fluence is high, the soot sublimation model will take some errors for measurement of soot particle size [63, 64]. Besides, nobody studies how to account for the effect of particle aggregation on soot sublimation.

4) Is Rayleigh-Debye-Gans approximation acceptable for LII application? When soot particles aggregate, the size of soot aggregates is not suitable for Rayleigh approximation. In this situation, LII may use Mie theory instead of Rayleigh approximation.

4.2 Emission computed tomography

At present, emission CT is used in measurements of 1-D axisymmetric flame. The challenge of this technique is simultaneous reconstruction of distributions of temperature and soot volume fraction in asymmetric flames.

Actually, inverse radiative problem can radically come down to the entirely extremum problem of objective function constituted by the measured/desired parameters and the estimated/designed parameters [65, 66]. The solution of inverse radiative problem includes optimization methodology [67] and regularization methodology [68]. Regularization methodology works by generating solutions to a sequence of well-posed problems that approximate the original ill-posed problem. Well-posed problems that are closely related to the original ill-posed problem have solutions that satisfy the ill-conditioned set of equations with a very small residual. It has been used to reconstruct temperature distributions [69]. The basic governing equation for the radiative energy transport process in participating medium is an integral and differential equation and the semi-transparent medium is emitting, absorbing, scattering in whole space. Inverse radiation analysis is generally concerned with the simultaneous determination of distributions of various types of radiative parameters, which makes the solution of inverse radiative problem become very complicated [65, 66].

At present, to decrease the complexity of inverse radiative problem, the radiation model is simplified. For example, in refs. [23, 52–58], the emission of axisymmetric ethylene-air diffusion flames were considered while the scattering and self-absorption were neglected. In refs. [24, 59–62], the scattering of flames were neglected. However, soot scattering measurements in visible spectrum showed that scattering to extinction ratio for ethylene-air flame was about 20% [70]. Therefore, the simplified radiation models neglecting scattering and self-absorption may take some errors. Moreover, the temperature and soot volume fraction in hydrocarbon-air flames are strongly coupled in the radiative heat transfer. For the combined reconstruction of the two types of parameters, the objective function is commonly a multimodal function, and the solution of entirely extremum problem is very difficult [65–67].

It should be mentioned that monochromatic radiative intensity images were only used in reconstruction in previous researches. In fact, temperature and emissivity images obtained by the two-color method, as a necessary supplementation to intensity images, can be used to simultaneously reconstruct temperature and soot volume fraction. In refs. [61, 62], a decoupled reconstruction method which combined optimization methodology and regularization methodology was used to simultaneously reconstruct distributions of temperature and soot volume fraction from flame temperature and emissivity images. Simulation investigation proved that the decoupled reconstruction method is of good accuracy and robustness. And the particle swarm optimization (PSO) was able to find a global optimum solution or a good approximation of the solution to the multimodal function [71]. There were a few reports concerning the application of PSO to inverse radiation analysis in recent years [66, 72–75]. In ref. [76], the preliminary research was done in simultaneous reconstruction of temperature and soot volume fraction by PSO.

5 Conclusion

Soot, which is produced in fuel-rich parts of flames as a result of incomplete combustion of hydrocarbons, is the No. 2 contributor to global warming after carbon dioxide. Developing soot measurement techniques is important to understand soot formation mechanism and control soot emission. The measurement principle and application cases of various soot measurement techniques have been reviewed in detail. The characteristics of these measurement methods were analyzed. In the development of soot measurement, LII and emission CT should be improved to realize the online measurement of multi-dimensional distributions of temperature, soot volume fraction, soot particle size and other physical and chemical parameters in hydrocarbon-air flames. Soot measurement techniques should be developed to meet both small flames in laboratories and large-scale flames in industrial combustion devices. Finally, in some special situation, such as high-pressure flames and zero gravity or micro-gravity flames, soot measurement also should be provided.

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