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Laser spectroscopy of thermal plasma

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Abstract

Thermal plasma, due to its applications, is a research field of great importance, but reliable diagnostics of such plasma remains a challenging task. Spatially resolved methods, which provide local values of plasma parameters, are crucial for understanding the underlying physics. This can be achieved using pump–probe techniques. Two methods applicable and useful for thermal plasma diagnostics—four-wave mixing and scattering of laser beams—are discussed in this paper. Experimental examples of their application, namely four-wave mixing in argon arc plasma and scattering of laser light by laser-induced plasma, are presented.

Keywords: thermal plasma, laser-induced plasma, plasma diagnostics, four-wave mixing, Thomson scattering

(Some figures may appear in colour only in the online journal)

1. Introduction

Thermal plasmas are those in which the heavy species temperature is approximately equal to the electron temperature. Due to a large difference in mass, electrons and heavy particles are weakly thermally coupled and the isothermal balance can reach equilibrium if elastic collisions between these two types of plasma constituents are sufficiently frequent. This requirement implies high electron density, pressure, temperature and, as a consequence, important ionization degree. Typically, the electron density is in the range $10^{22} - 10^{23}$ m$^{-3}$ while the temperature is in the range $10^4 - 10^5$ K \cite{1}. Thermal plasma is characterized by strong radiative emission, the spectrum of which consists of an intense continuous background superimposed by appreciably broadened spectral lines. They are systems with species distributed inhomogeneously and steep gradients of temperature. Isothermal equilibrium in the core is lost at colder and less ionized boundaries. All these features make thermal plasma diagnostics a special case.

Thermal plasmas can be created by electric fields in electric arcs, inductively coupled RF energy, microwave energy or laser energy. Transient mode operates often in pulsed modes on time scales of hundred $\mu$s or less. For instance, laser-induced plasmas experience a fast temporal evolution of their characteristic parameters, starting from plasma formation during the absorption of the laser pulse. In the presented review, discussion will be restricted to two cases: dc arc plasma as an example of stationary plasma and plasma induced by a pulsed laser as an example of transient plasma. They are important for applications—for instance for welding/cutting arc or laser-induced breakdown spectroscopy. For more detailed information the reader should refer to appropriate reviews, such as \cite{1}. Essential parameters for determination are electron density together with electron and heavy particle temperatures. These three parameters are those which determine the physics and chemistry of plasma. Discussion of diagnostic methods for their determination is a goal of this review. An ideal plasma diagnostic method should fulfil several requirements: (i) high spatial resolution adequate to gradients of plasma parameters and composition; (ii) temporal resolution sufficient to follow variation of the plasma state (the critical property for transient plasma); (iii) interpretation of the experimental results should be free from any assumptions about the plasma state; (iv) the plasma state should not be disturbed by the measurement—the method should be non-intrusive.

Unfortunately, there is no such method that could meet all these requirements, and existing methods are a compromise on these requirements. For a strongly inhomogeneous object such as thermal plasma, spatial resolution, which provides local values of plasma parameters, is a crucial property of the method and this aspect will be discussed here.

The most simple and most widely used method to diagnose plasma is emission spectroscopy—a passive one, so non-intrusive. However, it is not spatially resolved. Only
the intensity integrated along the line of sight can be measured and then determination of local values needs some numerical procedures, such as Abel transformation which is more or less applicable. Moreover, possible re-absorption in the outer, cooler zones of the plasma makes reliable inversion impossible. Active methods based on measurements of transmitted radiation are not only more or less intrusive but also naturally non-local methods—the signal is integrated along the probe beam.

The spatially resolved methods should be based on the pump–probe approach, where the measured signal originates from the intersection of two directions—pump and probe. The first method that naturally comes to mind, is laser-induced fluorescence in various variants [1]. It is commonly and successfully used in studies of low-pressure plasmas. However, in the case of high-density plasmas the optical signals are strongly attenuated by non-radiative, collisional processes and disappear in the midst of background fluctuations. The only methods to consider as spatially resolved and useful for thermal plasma diagnostics are four-wave mixing and scattering of laser light. These methods are used in diagnostics of low-temperature, low-density plasmas. An excellent review can be found in [2]. However, thermal plasma differs in several aspects from low-density plasma, which makes the application of this method a special challenge.

2. Four-wave mixing

Four-wave mixing is a coherent third-order nonlinear process, where three laser beams, two pump beams (1 and 2) and one probe beam (3), overlap in the medium under investigation. By a nonlinear interaction with the medium, a fourth beam called signal beam (4) is generated. Its properties are defined by energy and momentum conservation [2]:

\[
\omega_1 + \omega_2 = \omega_3 + \omega_4, \quad \mathbf{k}_1 + \mathbf{k}_2 = \mathbf{k}_3 + \mathbf{k}_4,
\]

where \(\omega_i\) and \(\mathbf{k}_i\) denote frequencies and wave vectors of the beams involved, respectively. The latter relationship is known as the phase matching condition. The well-known four-wave mixing process is coherent anti-Stokes Raman scattering (CARS), where two lower levels \((\alpha \text{ and } \beta)\) are real and \(\omega_1 - \omega_3 = 2\pi (E_{\beta} - E_{\alpha})/\hbar = \omega_{AB}\).

CARS is applied in various variants mainly to molecular species in low-temperature plasmas to determine ro-vibrational population distributions [2]. In thermal plasmas molecules are fully dissociated and it is difficult to find a pair of levels suitable for measurements. A promising variant of the four-wave mixing process is the resonantly enhanced, degenerate phase conjugate mixing case, which involves two real atomic levels; it means that \(\omega_1 = \omega_2 = \omega_3 = \omega_4 = \omega_{\text{AB}} = \omega_{\text{L}}\). This variant involves two counter-propagating laser pump beams \((\mathbf{k}_1 = -\mathbf{k}_3)\) crossed by the probe beam. The signal beam is thus phase conjugated to the probe beam \(\mathbf{k}_3 = -\mathbf{k}_4\), which means that it propagates collinearly with the probe beam but in the opposite direction. To separate them, pump beams have linear and mutually perpendicular polarizations and as a consequence, signal and probe beams have also orthogonal polarization and can be easily separated (see figure 1(a)). Plasma is probed with high spatial resolution limited by dimensions of the laser beams and the intersection angle between them. Moreover, since the signal beam is conjugated with the probe beam, it is emitted at a very small solid angle and the signal can be relatively easy to distinguish from strong emission of the plasma. Additionally, as the process is resonant it does not require intense beams so it can be treated as nearly non-intrusive.

There are several theoretical models of the phase conjugate degenerate four-wave mixing (PC-DFWM) in the literature [3, 4]. The model suitable for thermal plasma diagnostics was developed by Dziere\'zga et al [6, 7]. The derived model is applicable for any laser intensity, taking into account the configuration of the laser beams, the velocity of atoms and interaction with plasma.

Calculation of the PC-DFWM signal beam intensity consists of two steps: (i) calculation of the total polarization of the medium irradiated with three beams and (ii) calculation of the amplitude of the PC-DFWM signal for the given laser frequency \(\omega_L\) at a large distance from the interaction region.

To calculate the total polarization of the system irradiated with laser beams, the time-dependent density-matrix equations for the two-level open system have to be solved [5], namely

\[
\begin{align*}
\dot{\rho}_{12} &= -(i\omega_{12} + \gamma) \rho_{12} + i\hbar^{-1}V_{12} (\rho_{11} - \rho_{22}) , \\
\dot{\rho}_{11} &= \Lambda_1 - \gamma_1 \rho_{11} - i\hbar^{-1} (V_{12}\rho_{21} - V_{21}\rho_{12}) , \\
\dot{\rho}_{22} &= \Lambda_2 - \gamma_2 \rho_{22} + i\hbar^{-1} (V_{12}\rho_{21} - V_{21}\rho_{12}) .
\end{align*}
\]

The diagonal-matrix elements \(\rho_{11}\) and \(\rho_{22}\) are proportional to the population of levels 1 and 2 while the off-diagonal elements \(\rho_{12} = \rho_{21}^*\) describe the optical fluxes.
coherences between levels 1 and 2. The interaction term, \( V_{12} \), in the rotating wave approximation is given by

\[
V_{12}(r, t) = -\frac{1}{2} \mu_{12} \sum_{n=1}^{3} E_n \exp \left[ -i (\omega_n t - k_n \cdot r) \right],
\]  

(3)

where \( \mu_{12} \) stands for the dipole moment of the atom and \( E_n, \omega_n \) and \( k_n \) are the amplitude of the electric field, frequency and wave vector of the \( n \)-th beam, respectively. The decay rate of the total coherence \( \gamma \) and the average decay rate of the population \( \Gamma \) are given by the following relationships:

\[
\gamma = \frac{1}{2} (\gamma_1 + \gamma_2 + \gamma_{el}), \quad \Gamma = \frac{1}{2} (1/\gamma_1 + 1/\gamma_2)^{-1},
\]  

(4)

where \( \gamma_{el} \) is the part of the dephasing rate due to elastic collisions while \( \gamma_1 \) and \( \gamma_2 \) are the population decay rates of levels 1 and 2 due to inelastic collisions. In the absence of laser beams, both levels are populated at pump rates \( \Lambda_1 \) and \( \Lambda_2 \) and the population difference \( \Delta N_0 = \Lambda_2/\gamma_1 - \Lambda_1/\gamma_1 \). The dephasing rate \( \gamma \) corresponds to the electron impact broadening, which is directly proportional to the electron number density \( n_e \). The interaction of the atom with surrounding particles not only affects its decay rates but also modifies its transition frequency, which is included as the Stark shift \( d_\delta \) and broadening \( w_\delta \) of the levels involved (figure 1(b)). The polarization per atom is expressed as

\[ P(r, t) = \mu_{12} (\rho_{12} + \rho_{21}). \]

By analogy with holography, the PC-DFWM signal can be understood as diffraction of one of the beams on the spatial grating of the refractive index produced by the two other beams. In general, two types of gratings can be simultaneously induced: population gratings and coherence gratings. The induced gratings can be easily destroyed by atomic motion, which is strongly dependent on the geometric configuration of the employed laser beams. In the investigated PC configuration with small \( \theta \), only the grating arising from interference between the forward pump and probe beams is relevant as its period is much larger than the period of the other two gratings. Furthermore, the contrast of the grating is the largest if these beams are of equal intensity. In other words, the best experimental conditions are a small angle between the forward pump and probe beams and equal intensities of all three beams. It means that only the approach proposed by Brattlæan et al [4] is appropriate. The amplitude of the PC-DFWM signal, \( E_i \), for the given laser frequency \( \omega_0 \), at a large distance from the interaction region, in the dipole approximation and for the optically thin medium is given by the integral of the \( P_2 \) polarization amplitude over the whole interaction region, while \( P_2 \) is that component of the polarization \( P(r, t) \) which satisfies the phase matching condition defined in equation (1). The influence of the laser-beam temporal pulse shape on the PC-DFWM signal characteristics can be derived assuming that relaxation times \( \gamma^{-1}, \Gamma^{-1} \) are much shorter than the laser pulse duration. Not going into details of the full solution of the problem that the reader can find in original papers [6, 7], for practical purposes asymptotic behaviour of the signal is important. In the limit of non-saturating laser beams of equal intensities (\( I_1 = I_2 = I_3 = I \)), the spectral profile of the signal is proportional to the third power of the Lorentz profile:

\[
I_x \propto \mu_{12}^2 \Delta N_0^2 \frac{\Gamma}{\gamma} \left( \frac{S}{1 + \delta^2} \right)^3,
\]  

(5)

where \( \delta \) is the normalized detuning, which can be expressed as

\[ \delta = (\omega - \omega_0 - d_\delta)/w, \]

(6)

where

\[ w = w_\delta (1 + 4\delta)^{1/2} \]

(7)

is the power broadened width of the investigated line. In the above formula \( \delta \) stands for saturation parameter \( I/I_{sat} \), where \( I_{sat} = \varepsilon_0 c^2 \gamma_0 \Gamma (\mu_{12})^{-2}/2 \) is the saturation intensity [5], while \( w_\delta \) and \( d_\delta \) denote Stark width and shift, respectively.

At the saturation limit (\( S \gg 1 \)), no detuning, the intensity of the signal beam is characterized by the power of the laser intensity with the exponent dependent on the temporal shape of the laser pulse:

\[ I_x \propto \Delta N_0^2 \frac{\Delta}{\gamma} S^p. \]

(8)

For typical shapes of laser pulse, \(-0.1 < p \leq -1.0\). Instead of cumbersome calculations appropriate for a particular pulse shape, it is better to determine exponent \( p \) experimentally as asymptotic behaviour of the saturation curve \( I_x = f(S) \) at a fixed point in the plasma as shown in figure 2. It was obtained in an experiment similar to that presented in [6]. Plasma was created by a transferred arc burning in argon at atmospheric pressure and an arc current equal to 200 A. Measurements were performed for the 4p[1/2]1−→3s[3/2]Ar I transition (696.54 nm). Three beams of equal intensities involved to perform PC-DFWM were delivered by a tuneable dye laser pumped by the second harmonic of a Nd:YAG laser with 10 Hz repetition rate. The dye laser was operated with LDS698 dye and provided 8 ns pulses with a spectral bandwidth less than 2.5 GHz and of 10 mJ energy which can be reduced using an optical attenuator. The output beam of the dye laser was split into three beams of equal intensities with the backward pump beam polarized perpendicularly to the other two beams. Two counter-propagating pump beams intersecting with the probe beam at an angle of 300 mrad determined the spatial resolution of the method to be at least 0.2 mm longitudinally and 0.4 mm transversally. The prism monochromator discriminated the signal from the background of plasma light while the

Figure 3. PC-DFWM spectral profiles of 696.5 nm Ar I line determined at low laser intensities at distances of 2.5 mm (a) and 7.0 mm (b) away from the cathode tip on the arc axis burning in argon at an arc current equal to 200 A. The determined Stark shifts amount to $-1.34(1)$ and $-0.67(1)$ cm$^{-1}$, respectively. ● experimental PC-DFWM signal intensity; —— theoretical PC-DFWM spectral profile fitted according to equation (5); - - - - calibration signal intensity from the hollow cathode discharge.

Figure 4. Signal beam intensity $I_4$ along a TIG arc burning in argon at atmospheric pressure (a) and the corresponding temperature distributions (b) for different arc currents.
of scattering centres. As far as TS is concerned, the situation is more complex because in thermal plasma scattering is partially collective. Electrons in plasma are exposed to the fluctuating electric micro-field, thus influenced by collective oscillation. Electron fluctuations occur at low frequencies due to ion acoustic waves and at high frequencies near that of the electron Langmuir plasma wave. The scattering form factor reflects these two types of collective motion of electrons. It consists of a low-frequency structure called the ion feature, and a symmetric pair of broad satellites termed the electron feature. These satellites are separated from the laser frequency by $Δω = [2ω_{pe}/(ε_0m_e)]^{1/2}$, where $ω_{pe} = [ε_0n_e/(ε_0m_e)]^{1/2}$ is the plasma frequency.

Typically, the ion contribution is almost two orders of magnitude narrower than the electron contribution in the frequency or wavelength domain so that they are almost completely separated. The deconvolution of the ion feature is a very challenging task because of its narrow width and overlapping with the Rayleigh and stray-scattered laser light. The ion feature, Rayleigh scattering form factor and apparatus profile of the typical grating spectrometer are of comparable magnitude. The collective or non-collective character of TS is governed by the scattering parameter $α = 1/(kλ_D)$, where $λ_D = (ωp_eT_e/e^2n_e)^{1/2}$ stands for the Debye length and $k$ for the scattering wave vector equal to $4π sin(θ/2)/λ_L$, where $λ_L$ denotes the laser wavelength and $θ$ denotes the scattering angle. When $α ≪ 1$ the oscillations are damped and the scattering process is incoherent, in the opposite case $α ≫ 1$ scattering has a collective character (see figure 5). For thermal plasma, scattering is partially collective since even for a right angle $α$ is of the order of unity, which makes it different from low-pressure plasmas.

In general, the scattering form factor is a rather complex function of plasma parameters. Detailed discussion of the theory of TS is presented in [11] and in several textbooks, e.g. [12-13]. The shape of the form factor strongly depends on the scattering parameter $α$ and also on the plasma composition and temperature (figure 5). The electronic feature contains information about the electron density and electron temperature whereas the ionic feature contains information also about the ratio of the electron temperature $T_e$ and the heavy species temperature $T_i$ (figure 6). The latter is a very important property because methods sensitive to heavy particles’ temperature are very scarce.

TS was applied to arc plasma—a stationary one [8, 14-19]. But advantages and the power of the TS are more visible when applied to transient thermal plasma, for instance laser-induced plasma. The most important case is laser-induced breakdown spectroscopy—a method of atomic emission spectroscopy that uses laser-generated plasma as the hot vaporization, atomization and excitation source. As emission spectroscopy is used to diagnose plasma it is touched by all its shortcomings—spatial and temporal averaging. TS is then a good tool for supporting diagnostics and overcoming the troubles of emission spectroscopy. There are only a few studies investigating TS in laser-induced plasma [20-25].

The simplest case, suitable to fundamental studies, is the laser-induced breakdown in noble gas. The typical experimental setup for such experiments is rather simple. The chamber is filled with working gas at atmospheric pressure and continuously purged. One of the Nd:YAG lasers delivers a beam, which is focused on the centre of the chamber and generates the plasma. After laser pulse irradiation, the plasma plume expands and cools down in roughly a millisecond. The other laser delivers probe beams to be scattered in the plasma. The probe beam propagates perpendicularly and focuses on the centre of the chamber and continuously purged. One of the Nd:YAG lasers delivers a beam, which is focused on the centre of the chamber and generates the plasma. After laser pulse irradiation, the plasma plume expands and cools down in roughly a millisecond. The other laser delivers probe beams to be scattered in the plasma. The probe beam propagates perpendicularly and focuses on the region of plasma with some delay with respect to the first one. The scattered light is observed perpendicularly to the laser beams and is directed to the spectrometer equipped with a gated intensified charge-coupled device (ICCD) camera. Opening the entrance slit and setting the grating to zero-order detecting system can be used for plume imaging.

As an example, one can recall experiments presented in [23, 24]. Plasma was generated in argon by second harmonic of Nd:YAG ($λ = 532$ nm) laser pulses of $2$ kJ cm$^{-2}$ fluence and $6$ ns duration. The scattered beam of fluence equal to $18$ J cm$^{-2}$ was delivered by the second Nd:YAG ($λ = 532$ nm) laser orthogonal to the breakdown beam. Plasma images and scattering spectra were observed perpendicularly to both beams using a spectrograph (750 mm focal length and $1.005$ mm$^{-1}$ reciprocal dispersion) equipped with a gated two-dimensional ICCD camera consisting of 1024 $×$ 1024 pixels of $13 \times 13$ μm size and the shortest gate equal to $2$ ns. For other details the reader should see [23, 24].

Measurements performed using such an installation can provide an overwhelming amount of information. An example is shown in figure 7. The spectrum presented in this figure was obtained at the axis of the plasma plume at the axial
Figure 7. Typical experimental data obtained from plasma induced by a laser pulse of 2 kJ cm\(^{-2}\) fluence and 6 ns duration in argon 3 \(\mu s\) after breakdown. (a) Plasma image, the breakdown beam is marked by a large green arrow while the scattered beam is marked by a dashed yellow line. (b) Thomson spectrum obtained by scattering of the second harmonic of a Nd:YAG laser (\(\lambda = 532\) nm) propagating along the \(z\)-axis at \(y = 0\). (c) Central part of the TS spectrum presented in different pseudo-colour scale. (d) Cross-section of the TS spectrum along the \(z\)-axis at \(\lambda = 532\) nm. (e) Cross-section of the TS spectrum at \(z = 0\). Theoretical profile fitted to experimental electron feature points gives \(n_e = 1.23 \times 10^{23} \text{ m}^{-3}\) and \(T_e = 19,700\) K.

position close to the point of maximum emissivity 3 \(\mu s\) after breakdown. It consists of two crescent shaped structures symmetrically separated with respect to the laser wavelength. This is the electronic feature of the TS and its shape is due to strong variation of the electron density and electron temperature occurring along the probe beam. The central part is appreciably brighter and is shown in different pseudo colour scales at the right. It consists of the ionic feature of the TS and Rayleigh scattering. Outside the hot plume there is typical pure Rayleigh scattering. At the cross-section of laser frequency, outside the hot and luminous fireball, there is a clearly visible shock front of the shock wave created by the spark ignition and detached from the plasma fireball. The measured spectral distribution in one row (it means at some position along the \(z\)-axis) can be fitted to the theoretical shape of the electronic feature and in this way the electron density and electron temperature are simultaneously determined, namely \(n_e = 1.23 \times 10^{23} \text{ m}^{-3}\) and \(T_e = 19,700\) K. It should be emphasized that these plasma parameters were derived without hypothesis concerning the equilibrium state of the plasma. Repeating the procedure for each row distribution \(n_e(z)\) and \(T_e(z)\) can be easily obtained. In the presented experiment, Thomson spectra were registered from 100 ns up to 10 \(\mu s\) after breakdown, at a fixed point on the axis of the plume, and the results are shown in figure 8. It should be stressed that such a result is difficult to obtain with other methods.

Profiting from Rayleigh scattering, temporal and spatial evolution of the shockwave can be investigated [24]. This shock wave is a result of explosion occurring during breakdown. The shock wave is detached from the luminous region, called the ‘fireball’. The detachment occurs at the instant when the temperature behind the shock front decreases to values insufficient for noticeable ionization of the gas. One should distinguish between shock wave parameters and ‘fireball’ parameters since their evolution is independent of each other. Note that such measurements are usually performed by shadowgraphy. In the scattering experiment, also plasma parameters in the fireball are simultaneously determined with the shock front position.

Besides measurements of the electron density and temperature, TS can also serve for measurements of the ion temperature, which is important for verification of the existence of isothermal equilibrium in laser-induced breakdown spectroscopy (LIBS) plasmas. This information is contained in the ion part of the spectrum. The central peak around the laser frequency arises due to the Rayleigh scattering and the ionic feature of the TS. Under conditions of LIBS plasmas, their spectral range is comparable. They overlap, but if the spectral resolution of the apparatus is satisfactory, the distinction of these spectra is possible. Both profiles, Rayleigh scattering and the ionic feature, depend on heavy particles’ temperature, while the ionic feature depends
on the electron density and temperature also. These two parameters can be derived from the electronic feature.

The registered profile around the laser frequency is a convolution of the apparatus profile and scattering signal. The apparatus profile $A(\Delta \lambda)$ can be approximated by the Rayleigh scattering profile far away from the plasma plume. Thus, the decomposition of the central profile can be performed by fitting to the measured points a function containing only three free parameters—two amplitudes and ion temperature:

$$I(\Delta \lambda) = \int A(\Delta \lambda - \Delta \lambda') [a S_R(\Delta \lambda', T_h) + b S_i(\Delta \lambda', T_e, T_h)] d(\Delta \lambda').$$  \hspace{1cm} (9)

An attempt to perform such measurements can be found in [26] for helium plasma at a time interval ranging from 20 to 800 ns after the breakdown.

The electron density decreased from $7.8 \times 10^{23}$ at 20 ns to $2.6 \times 10^{22} \text{ m}^{-3}$ at 800 ns after the breakdown pulse. In the same time, the measured electron temperature dropped from about 95 900 to 10 350 K and largely exceeds the heavy particle temperature, which decreased from 47 000 to 4100 K. There exists a large discrepancy between electron and heavy particle temperatures, which indicates two-temperature plasma, out of equilibrium.

4. Conclusion

In conclusion, it can be stated that laser scattering methods are very useful in thermal plasma studies not only because they have high temporal and spatial resolution, but also since all of their results do not rely on assumptions about the equilibrium state of the plasma. The most important parameters—electron density, electron temperature and heavy species temperature—can be deduced from scattering spectra. In particular, the heavy species temperature is extremely difficult to obtain with other methods.

References

[26] Dzierżega K et al 2013 Appl. Phys. Lett. 102 134108