



Spatially and temporally resolved fs/ps CARS measurements of rotation-vibration non-equilibrium in a CH₄/N₂ nanosecond-pulsed discharge

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Abstract: Due to concerns about climate change, there is significant interest to establish CH₄ lean burn engines or convert it to valuable industrial chemicals using non-equilibrium plasmas. To quantitatively understand the dynamics and chemistry of plasma discharge in CH₄ fuel mixtures, it is necessary to obtain time and spatially resolved data of key parameters such as the CH₄ concentration and degree of rotation-vibration non-equilibrium. Rotational fs/ps CARS was used to simultaneously measure rotational and vibrational temperatures of a pin-to-pin 40% CH₄/60% N₂ nanosecond-pulsed discharge at 60 Torr, while the CH₄ concentration was measured by vibrational CARS. The measurement region was 2 mm along the electrode axis, within 150 μm of the cathode surface. Gradients in N₂ rotational and vibrational temperature and CH₄ number density were observed to evolve in time and space. The vibrational temperature peaked above 6000 K, 100 μs after the voltage pulse, and the majority of CH₄ consumption occurred during the voltage pulse. Additional CH₄ consumption along with rapid heating occurred during the first 2 μs of the afterglow, indicating a role of electronically excited N₂ quenching in dissociation of CH₄.

1. Introduction

To prevent global climate temperatures from exceeding 1.5°C, there has been much interest in electrifying industrial chemical processes such as CH₄ reforming and lean burning CH₄ flames for gas turbine engines by using non-equilibrium plasmas [1–6]. However, quantitative understanding of the important reaction pathways in CH₄ reforming has not been achieved due to the lack of model validation metrics measured *in-situ* for even CH₄/N₂ mixtures. Previous work in CH₄/N₂ plasmas were mostly focused on studying chemical processes in Titan’s atmosphere [7] and synthesizing materials such as carbon nitride coatings [8]. For model validation, a pulsed discharge is preferable to define a start and end to the discharge as well as to study chemistry in the afterglow. While, pure N₂ or air pulsed discharges have been well-studied and characterized [9–12], only a few studies have explored CH₄/N₂ pulsed plasmas for methane reforming purposes [3,13] or characterized the non-equilibrium state of the plasma [14]. From past numerical

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simulations, the major dissociation pathway for CH₄ is either through direct electron impact or collisions with electronically excited nitrogen molecules such as N₂(A) [3,13]. However, it is also known that electronic-vibrational (E-V) coupling can be a notable source of vibrational energy in N₂ nanosecond pulsed pin-to-pin discharges [15,16]. Unfortunately, this process was not considered by [3,13], so it is neither clear how quenching of N₂ electronic levels by CH₄ affects the evolution of the rotation-vibration non-equilibrium in N₂, nor is it clear whether there are additional reaction pathways for CH₄ in highly vibrationally excited N₂ mixtures.

Hybrid fs/ps coherent anti-Stokes Raman scattering (fs/ps CARS) is a powerful laser diagnostic capable of probing gas temperature and species concentrations on the picosecond time scale with tens of microns of resolution [17–21]. A methodology for retrieving vibrational and rotational temperatures simultaneously from spatially-resolved 1-D rotational fs/ps CARS images was recently developed [22]. This method takes advantage of the red-shift of rotational energy levels of vibrationally-excited molecules to infer the vibrational temperature along with the rotational temperature. This allows for spatially and time-resolved measurements of rotation-vibration non-equilibrium from one measurement. Additionally, a broadband femtosecond pulse produced from self-phase modulation in a hollow-core fiber was used as the pump/Stokes beam. The generation of Raman coherences up to the C-H stretch of CH₄ was achieved like in past works [19,23]. Therefore, both rotational and vibrational temperatures as well as the CH₄ Q-branch could be measured using the same setup.

In this study, to understand the non-equilibrium dynamics and evolution of the CH₄ number density in a CH₄/N₂ nanosecond-pulsed pin-to-pin discharge, the time-resolved 1-D rotational and vibrational temperatures and CH₄ number densities were measured using fs/ps CARS.

2. Experimental Methods

The detailed experimental setup description and theory behind the detection method is in [22] and only a brief overview will be given here. Hybrid fs/ps CARS takes advantage of the broadband nature of femtosecond laser pulses to probe multiple Raman transitions simultaneously, while the non-resonant background is avoided with a delayed spectrally-narrow probe pulse. In this study, two-beam phase matching is used where the both the pump and Stokes photons come from a single laser source [24]. The principle behind detecting vibrational and rotational temperatures simultaneously from the rotational is given by equations 1-3:

$$F(v, J) = B_v(J(J + 1)) - D_v(J^2(J + 1)^2) \quad (1)$$

$$B_v = B_e - \alpha \left(v + \frac{1}{2} \right) + \gamma \left(v + \frac{1}{2} \right) \quad (2)$$

$$D_v = D_e + \beta \left(v + \frac{1}{2} \right) \quad (3)$$

where F is the rotational energy of an N₂ molecule in a state (v, J) with v and J being the vibrational and rotational energy levels, B_v and D_v the vibrational energy level-dependent rotational and centrifugal constants, B_e and D_e are the equilibrium rotational and centrifugal constants, and α , β , and γ are rotation-vibration coupling constants. From equations 1-3, the rotational energy levels exhibit a red-shift with increasing vibrational energy due to the decrease in B_v and increase in D_v . This vibrationally-induced shift can be on the order of 1 cm⁻¹. For molecules in rotation-vibration non-equilibrium, molecules in the excited vibrational states will appear at these shifted wavelengths. Such shifts have been detected before using spontaneous Raman scattering and CARS [25,26]. The shifts can be seen in Fig. 1, where the overall envelope convolution of each of

the peaks with the spectrometer instrument function. Therefore, even without enough resolution to resolve each peak, a one-sided broadening can be detected, which can then be related to the distribution of the vibrational states and its associated vibrational temperature.

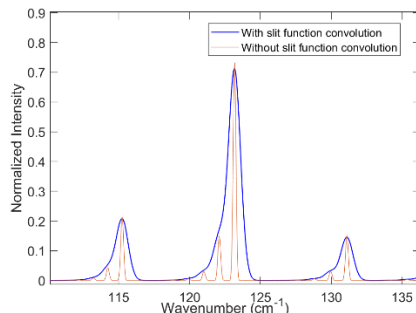


Figure 1. Modelled rotational spectrum at $T_r = 400\text{K}$ and $T_v = 4000\text{K}$. The underlying peaks are the fully-resolved shifted vibrationally-excited N_2 lines, while the overarching envelope is the total convolution of the spectrometer instrument with each of the peaks.

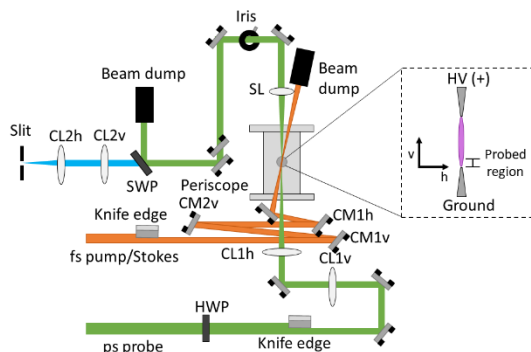


Figure 2. Experimental setup for fs/ps CARS imaging [22]. CM1h, CM1v, CM2v: concave cylindrical mirrors with $f = 400$ mm, 400 mm, and 200 mm. CL1v, CL1h, CL2v, CL2h: plano-convex cylindrical lenses with $f = 300$ mm, 400 mm, 400 mm, 75 mm. SL: spherical plano-convex lens with $f = 400$ mm. SWP: short wave pass.

The fs/ps CARS setup is shown in Fig. 2 and is similar to the setup in [19]. The pump/Stokes photons were generated by focusing 1.5 mJ of a Ti:Sapphire regenerative amplifier into a hollow core fiber (Femtolasers). The output of the fiber was a compressed laser pulse with a pulse width of less than 7 fs and 0.6 mJ of total pulse energy. A picosecond laser with a pulse width of 65 ps and pulse energy of 6 mJ operating at 20 Hz was used as the probe beam. The oscillator of the picosecond laser was phase locked to the oscillator of the femtosecond laser. Knife edges created sharp edges on the incoming beam profiles. The beams were then imaged to the probe volume which reduced light scattering off the cathode surface. The beams were cylindrically focused into sheets and crossed at a 5-degree angle in a 2-beam phase matching configuration. The resulting CARS signal traveled with the probe beam and was separated using an angle-tuned short wave pass filter. The CARS signal was then imaged onto the slit of the spectrometer (Horiba) and detected with a CCD camera (Andor) water-cooled to -80°C . The plasma was generated by applying a 4 kV, 500 ns pulse from a high voltage switch to the positive electrode (DEI). A 220 Ohm resistor was connected in series between the anode and the high voltage switch to limit the current. The voltage and current traces are shown in Fig. 3 with and without breakdown. The pulses

were applied at 20 Hz and were synchronized with the picosecond laser via a delay generator. CH₄ densities were measured by rotating the grating to the appropriate Raman shift and the signal was integrated across Q-branch region. Measurements at room temperature were taken periodically to serve as a number density calibration.

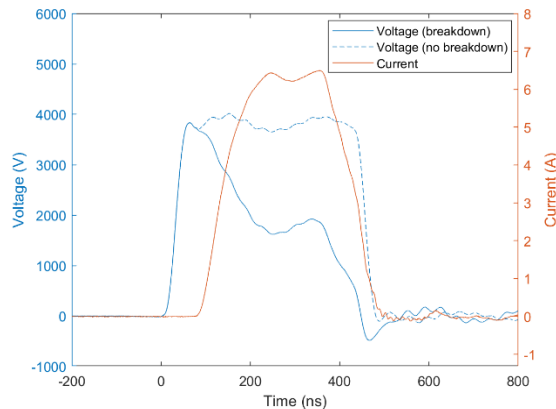


Figure 3. Measured voltage with and without breakdown as well as the conduction current.

3. Results and Discussion

The 1-D time-resolved profiles of the vibrational temperature, rotational temperature, and the CH₄ number density are shown in Fig. 4 and 5. One of the immediately visible trends is that around 100 μ s, the vibrational relaxation becomes dominant, with the vibrational temperature decreasing rapidly with the rotational temperature increasing by several hundreds of K. This is more clearly visualized in Fig. 6 by picking a single spatial location, 1500 μ m from the cathode, and plotting the time-resolved responses of the three measured parameters. At time scales greater than 100 μ s, conduction and diffusion work to smooth the spatial distribution of the rotational temperatures. Additionally, there are strong gradients in the vibrational temperature along the axis of the cathode, reminiscent of a DC glow discharge [27]. This could be possible, since the 500 ns voltage pulse width is much longer than the time to complete the gap, which can occur on tens of ns timescale or faster [28]. Therefore, a transient glow discharge could form, and if the voltage pulse was kept on for longer, transition to an arc would occur [29,30]. For 60 Torr of N₂ and 220 Ohm resistance, the time before the glow-to-arc transition is on the order of a few μ s [30]. If we assume that a transient glow has formed, then the region of high vibrational excitation from 600 to 1000 μ m corresponds to the negative glow region, where there is a higher electron number density. This would lead to more collisions with neutral molecules and more vibrational excitation through electron-impact. Additionally, this would imply that the cathode sheath thickness is 200-300 μ m thick which would be unlikely due to the high conduction current (\sim 6 A maximum). Further investigation is required to correlate the discharge structure with the vibrational temperature spatial distribution.

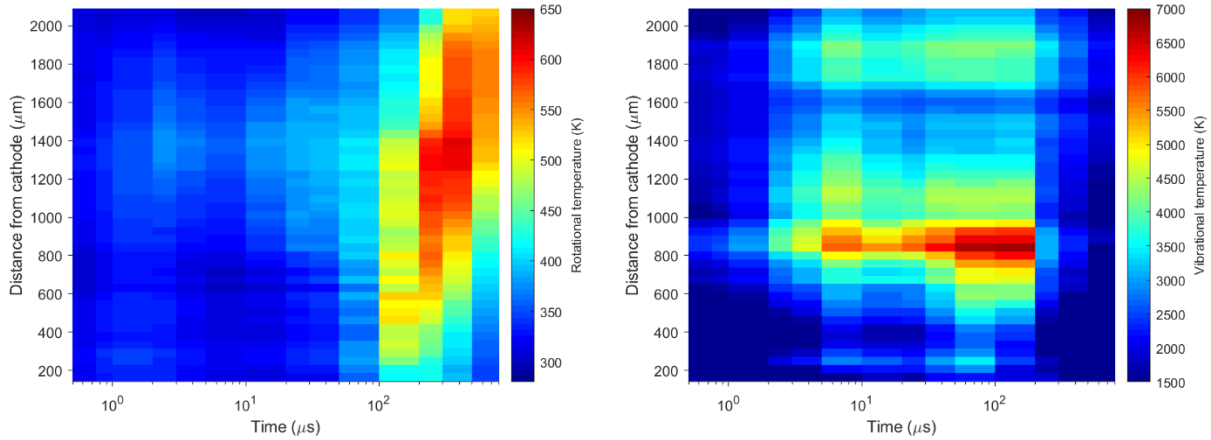


Figure 4. 1-D time-resolved profiles of the rotational (left) and vibrational (right) temperatures. The color bar denotes the temperature at the time marked by the left edge of each cell.

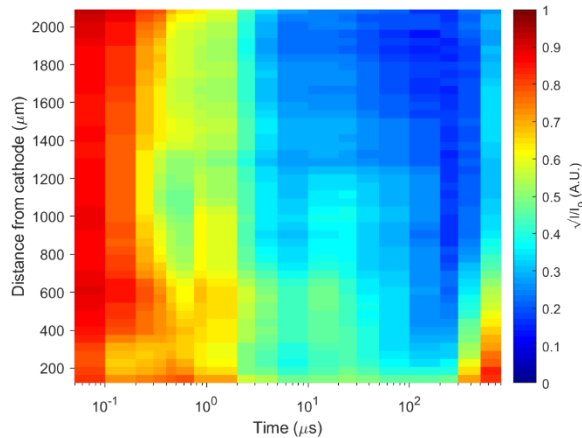


Figure 5. 1-D time-resolved profiles of the square root of the integrated CH₄ signal over the ν_1 Q-branch relative to ambient conditions. The color bar denotes the relative signal at the time marked by the left edge of each cell.

From Figure 6, the CH₄ number density decreased to 50-70% of its starting value within the first 500 ns. This corresponds to the time when the voltage pulse was applied. Afterward, the CH₄ number density fell even more, down to 20-30% of its original value after 10 μ s elapsed. At the same time, the rotational temperature did not increase by more than 20%. Therefore, the vast majority of this CH₄ number density decrease was due to dissociation of CH₄ rather than from temperature increase. During the pulse, the dissociation was most likely directly through electron-impact. However, on the μ s time scale, the electrons should not have sufficient energy to dissociate CH₄. It is known that electronically excited states of N₂ such as N₂(A) are efficiently produced in high E/N nanosecond-pulsed discharges and collisional quenching leads to “fast gas heating” [31,32]. In air plasmas, fast gas heating occurs due to heat release from N₂ electronic state quenching with O₂, which results in the production of two O atoms. These electronic states also possess sufficient energy to break a C-H bond in CH₄ at a similar rate to using O₂ as the collisional partner [13,31]. Therefore, the rise in temperature and decrease in CH₄ number density after the voltage pulse until 2 μ s was most likely due to this quenching. However, this does not explain the further decrease in CH₄ number density, after the end of the fast gas heating. This indicates that

there is an additional reaction pathway consuming approximately 15-20% of the total CH₄ that does not release significant amounts of heat. This decrease occurs during the time when the vibrational temperature increases by more than 1000K. However, between 5 μ s and 100 μ s, the CH₄ number density remains constant while the vibrational temperature reaches its maximum. Therefore, it is unclear what how the N₂ vibrational kinetics affect the CH₄ number density. Further investigation is required with a chemical kinetic model to fully understand these trends such as in [3].

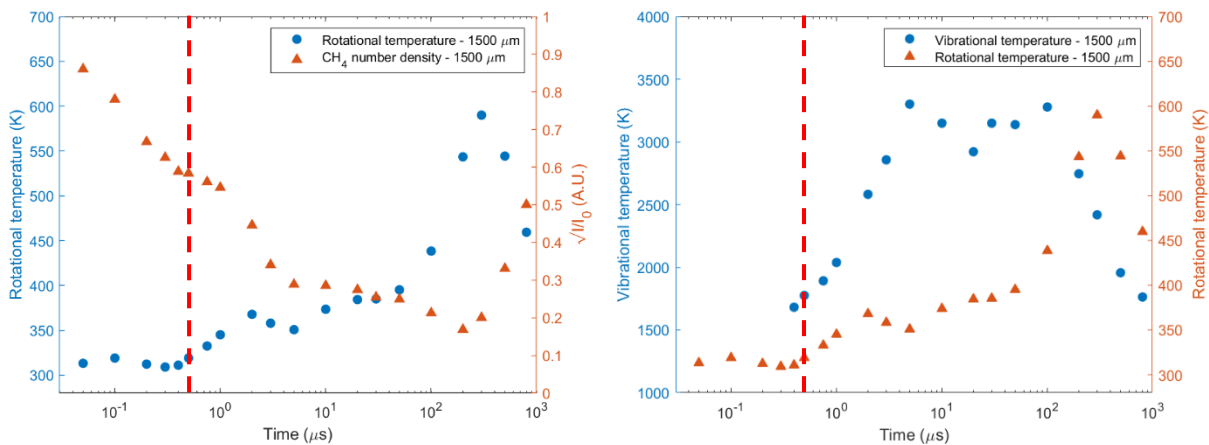


Figure 6. Time-resolved profiles of the CH₄ number density, rotational temperature, and vibrational temperature 1500 μ m from the cathode. The red dashed line marks the end of the pulse.

4. Conclusions

In this work, hybrid rotational and vibrational fs/ps CARS was applied to measure the one-dimensional time-evolution of rotational temperature, vibrational temperature, and CH₄ density in a nanosecond-pulsed CH₄/N₂ discharge. From the simultaneous rotational and vibrational temperature measurements, the rotation-vibration relaxation occurred from 100 to 300 μ s. From the 1-D vibrational temperatures, strong spatial gradients were observed. Since the applied voltage pulse was much longer than the time to connect the electrode gap, then a transient glow discharge could form after breakdown. Furthermore, from the CH₄ and rotational temperature time evolution, there was evidence for CH₄ dissociation from collisional quenching of electronically excited N₂. However, after the fast gas heating, even more CH₄ consumption occurred, without significant rotational temperature rise. This correlated with an increase in the N₂ vibrational temperature, but during the period of maximum N₂ vibrational temperature, the CH₄ number density was approximately constant. Additional modeling is required to understand how the vibrational kinetics influences the CH₄ conversion.

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