Promotion of methane ignition by the laser heating of suspended nanoparticles

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Abstract. The influence of laser heated iron and carbon nanoparticles on ignition of 20 vol% stoichiometric methane–oxygen mixture in argon was studied experimentally in shock tube reactor. The concentration of nanoparticles 0.3–2.0 ppm was measured by laser light extinction. The particles were heated by Nd:Yag laser pulse operated at wavelength 1064 nm. The ignition delay times were registered by increase of OH chemiluminescence and pressure rise. The temperatures of laser heated particles and their sizes were measured by laser induced incandescence technique. The significant decrease of ignition delay times were found at addition of iron particles heated by laser pulse to the combustible mixture at the temperatures less than 1400 K. Analysis performed has shown that the effect supposedly involves catalytic reactions of methane decomposition on the surface of heated particles and allowed estimating their effective activation energy.

1. Introduction

It is known that a radiative overheating of particles suspended in combustible gas mixture may lead to its ignition and detonation. Such an effect could affect the fire safety of large volumes where the radiation heat transfer became important for the explosion development; another practical application is the safety of fiber optic systems in combustible media, as the radiation from the damaged fiber could cause explosion. Proust [1, 2] experimentally investigated an ignition of dusty clouds as a result of combustible particles heating. Moore and Weinberg [3, 4] have shown that thin fibers being heated with CO$_2$-laser (pulse power was 20 W, the wavelength was 10.6 $\mu$m) could ignite ambient combustible gases and increasing of radiation intensity lead to ignition time decrease. It was concluded that suspended particles could promote ignition being heated by the intense radiation from the nearby flame front. As the ignition is critically depend on the energy absorbed by the particles, a spectral absorbance coefficient at given wavelength is the key particles feature. In work [5] Moore and Weinberg used blackened bodies for ignition promotion. Particles temperature measurements carried out by Adler et al [6] have shown that absorbance at 10.6 $\mu$m was noticeable bigger than one at 1.06 $\mu$m. Fiber structures capability to ignite combustible mixtures is the result of effective radiation absorbance and heat transfer to surround media due to high specific surface. Welzel et al [7] had studied ignition
promoted by iron and manganese oxide particles placed at the end of optical fiber. It was shown that minimal laser power resulting in ignition of methane–oxygen mixtures decreased as fiber diameter reduced down to 100 µm; further thinning had no effect. Dubaniewicz et al [8] studied the ignition induced by heated iron oxide and coal particles using the similar setup; non-combustible particles with good absorbance at radiation wavelength were discovered to be most effective. It was observed in [9,10] that critical laser energy necessary for ignition of hydrogen-air mixtures increases for larger, i.e. more massive particles. Stamatov et al [11] experimentally proved that methane–oxygen mixture could be ignited by a single chemically inert particle. Study [12] was devoted to investigation of butane-oxygen mixture ignition in presence of a wide variety of particles placed on support surface and irradiated by an infrared laser. Increasing of the laser intensity led to ignition delay time shortening for all studied particles. It was observed that ignition delay time is much more sensitive to particles properties such as chemical activity and spectral absorbance coefficient than to properties of support surface. Ignition was observed for many carbon-based materials such as graphite, fullerenes C_{60} and diamond powders, as well as silicon carbide and silicon oxide. It was shown that the particle size dramatically influences the ignition delay time. Smaller particles result in faster ignition due to higher surface to volume ratio.

All these results, though, were obtained for particles of micron size range placed at support surfaces. Influence of laser-heated particles of nanometer-range sizes suspended in gas media remains uninvestigated. Particularly the possible mechanism of the ignition promotion is unclear, as it may be thermal one, i.e. caused by local temperature rise, or catalytic, originated from active radicals formation on the particles surface. The goal of present work was the experimental investigation of the ignition of methane–oxygen mixtures at the presence of minor admixtures of carbon and iron nanoparticles, in particular, heated by laser radiation.

2. Experimental and modeling methods
Experiments were carried out in a stainless steel shock tube of a standard design with an inner diameter 50 mm consisting of a high pressure chamber (HPC) (length 1.5 m) and low pressure chamber (LPC) (3.0 m) separated by changeable membrane made of aluminum foil of 0.07 mm thickness. Initial pressure of investigated mixture in LPC was varied in range 0.08–0.30 bar. HPC was filled by the helium up to spontaneous membrane break resulting in incident shock wave (ISW) formation. After the reflection of ISW from the end plate of tube the reflected shock wave (RSW) compressed investigated mixture up to pressure \( P_5 \) and heated to temperature \( T_5 \) at which all measurements were carried out.

Shock wave velocity was measured using two piezoelectric calibrated pressure gauges PCB113B26 (PCB Piezotronics Corporation), which were placed at 45 and 139 mm or 15 and 109 mm distances from the end plates of different designs, one of which was used for synthesis experiments and the other one—for ignition experiments. Four optical windows installed in the investigated cross-section provided the possibility for optical measurements. Oscilloscope Tektronix TDS 2014B (200 MHz bandwidth) was used for signals registration.

Before every synthesis–ignition pair of experiments inner surface of shock tube was thoroughly cleaned with ethanol and the tube was pumped with a fore-vacuum pump to the pressure \( 4 \times 10^{-2} \) mbar. Used mixtures were prepared manometrically using argon and oxygen of 99.99% vol % purity (hereinafter, volumetric percentages are used), methane (99.99%), benzene (99.9%) and iron pentacarbonyl (99%, Sigma-Aldrich) and were allowed to homogenize during at least two hours before the experiment. Various values of temperature \( T_5 \) were obtained by varying initial pressure \( P_1 \). Ranges of experimental conditions were \( T_5 = 1050–2000 \) K and \( P_5 = 4.0–5.6 \) bar.

Used mixtures and experimental condition summarized in table 1. Mixtures 1–3 were used for the synthesis of iron and carbon nanoparticles behind the shock waves in the shock tube. Carbon nanoparticles were synthesized during the pyrolysis of mixtures of 1–2% \( \text{C}_6\text{H}_6 \) in argon behind
Figure 1. Scheme of the investigated section of shock tube equipped with the optical diagnostic of nanoparticles (i.e. laser extinction and laser-induced incandescence).

Figure 2. Typical extinction signal representing the formation of iron nanoparticles in mixture 1% Fe(CO)$_5$ + Ar.

the reflected shock waves at the temperatures $T_2 = 1800–2200$ K and pressures $P_5 = 4.0–4.6$ bar. Iron nanoparticles were synthesized during the pyrolysis of mixtures 1% Fe(CO)$_5$ in argon behind the incident shock waves at the temperatures $T_2 = 700–1000$ K and pressures $P_2 = 1.0–$
Figure 3. The volume fraction of nanoparticles entrained in the flow behind the shock wave in ignition experiments: 1—ignition parameters $T_5 = 1353$ K, $P_5 = 4.85$ bar, preliminary particles synthesis parameters $1\%\text{Fe(CO)}_5 + \text{Ar}$, $T_5 = 1268$ K, $P_5 = 5.04$ bar; 2—ignition parameters $T_5 = 1314$ K, $P_5 = 4.74$ bar, synthesis parameters $1\%\text{Fe(CO)}_5 + \text{Ar}$, $T_5 = 1862$ K, $P_5 = 4.43$ bar; 3—ignition parameters $T_5 = 1435$ K, $P_5 = 4.47$ bar, synthesis parameters $2\%\text{C}_6\text{H}_6 + \text{Ar}$, $T_5 = 1849$ K, $P_5 = 4.43$ bar.

1.7 bar, reflected waves additionally heated the particles causing their partial evaporation at the temperatures $T_5 = 1000–1900$ K and pressures $P_5 = 4.2–5.6$ bar. After the synthesis particles were deposited on shock tube walls. During the common procedure of membrane replacement and optical windows cleaning shock tube was opened and iron particles were exposed to atmosphere air and partially oxidized. Consequently LPC was filled with investigated mixture N4 and an ignition experiment was carried out. Particles deposited on shock tube walls were entrained in the flow behind the shock wave. Their concentration was measured by the laser extinction technique at 633 nm wavelength, and their sizes were determined using laser-induced incandescence method (figure 1).

The measurements of laser extinction allow detecting the presence of condensed nanoparticles as they cause absorbance and scattering of light. In given experimental conditions particles were definitely small in terms of Rayleigh limit. Gas laser JDSU He-Ne 1201-2 was used as a light source and photodiode PDA10A-ES (THORLABS) as a detector providing 10 ns time resolution. Detector was equipped with an interference filter $633 \pm 1$ nm. Typical signal of laser extinction for iron nanoparticles is shown in figure 2. Signal intensity was dropped rapidly after the incident shock wave propagation indicating the formation of condensed phase absorbing the laser radiation. Extinction decreased behind the reflected shock wave, supposedly due to partial evaporation of iron particles. The volume fraction of condensed particles varied in the range 0.3–2.0 ppm depending on the particle material, precursor mixture and temperature of synthesis (figure 3).
Figure 4. Typical signals of pressure gauge and OH* radiation detector during the ignition of stoichiometric 20%(CH\textsubscript{4} + O\textsubscript{2}) + Ar mixture.

Table 1. Experimental conditions.

| No | Mixture | \(T_5\), K | \(P_5\), bar |
|----|---------|------------|-------------|
| 1 | 1%Fe(CO)\textsubscript{5} + 99%Ar | 1000–1900 | 4.2–5.6 |
| 2 | 1%C\textsubscript{6}H\textsubscript{6} + 99%Ar | 2000–2200 | 4.0–4.2 |
| 3 | 2%C\textsubscript{6}H\textsubscript{6} + 98%Ar | 1800–1900 | 4.3–4.6 |
| 4 | 20%(CH\textsubscript{4}) + O\textsubscript{2} + 80%Ar | 1100–1900 | 4.4–5.1 |

The method of laser induced incandescence (LII) is based on fast heating of nanoparticles by a laser pulse and subsequent registration of their thermal radiation. LII signal intensity is proportional to the volume fraction of condensed phase and its decay time depends on particle size. Two-color LII measurements and detailed modeling of heating and cooling processes allow quantitative particles size measuring. Details of used LII technique could be found in [13].

Methane–oxygen mixture behind the RSW contained 0.5–2.0 ppm volume fraction of condensed iron or carbon particles which were heated by a laser impulse at 1.064 \(\mu\text{m}\) wavelength. Laser energy fluence was varied in the range 0.1–0.8 J/cm\(^2\). The heating occurred during 10 ns in 1–10 \(\mu\text{s}\) after shock wave propagating; as well as the heating time, such a delay was considered to be negligible in comparison with typical ignition delays in given mixture (100–1200 \(\mu\text{s}\)). The volume containing laser heated nanoparticles amounted to less than 1/60 of whole reacting volume.

The temperature dependence of the ignition delay time (also known as the induction time) is the key feature of the combustible mixture. OH radicals are the characteristic species for the indication of hydrocarbon ignition. During the experiments, an excited OH* chemiluminescence signal was recorded by a Hamamatsu H9307-03 photomultiplier module equipped with an interference filter (310 ± 5 nm) to determine the ignition delay times. In combustible mixtures, the energy release in ignition is quite abrupt and leads to a dramatic increase in the OH concentration in both ground and excited states. Thus, a rapid rise in OH* chemiluminescence
Figure 5. Signals of pressure gauge during the ignition of stoichiometric 20%(CH\textsubscript{4} + O\textsubscript{2}) + Ar mixture: 1—no nanoparticles \(T_5 = 1479\) K, \(P_5 = 4.51\) bar; 2—in presence of carbon nanoparticles, \(T_5 = 1480\) K, \(P_5 = 4.47\) bar; 3—in presence of iron nanoparticles, \(T_5 = 1456\) K, \(P_5 = 4.60\) bar.

was considered as the end of induction time. The exact ignition moment was determined as the intersection of the inflectional tangent line of the OH\textsuperscript* radiation plot with the time axis. The increase in pressure was simultaneously recorded by a pressure gauge.

Typical signals of pressure gauge and OH\textsuperscript* radiation detector are presented in figure 4. One should note that in the reference stoichiometric methane–oxygen mixture pressure remained constant during the induction time, while presence of carbon particles resulted in steady rise up to 20\%, and of iron ones—up to 35\% (figure 5).

Kinetic modeling was performed using ChemKin software [14] and thermodynamic data [15]. Since in the induction zone the typical rates of chemical reactions are much lower than the characteristic rates of gas dynamic processes behind a reflected shock wave, the classic approximation of a constant pressure reactor [16] was applied. Calculations performed with a time-step of 0.5 \(\mu\)s provided the time profiles of the concentrations of the investigated species. The ignition delay time was determined using the moment of rapid increase in OH concentration.

Kinetic mechanism GRI-Mech 3.0 [17] involving 325 reversible reactions of 53 species was used. It was shown in previous work [18] that it provides a good agreement with the experimentally obtained values of induction times of ignition of argon diluted methane–oxygen mixtures behind shock waves.

3. Results

Influence of laser-heated nanoparticles was investigated in a set of experiments carried out in non-admixtured methane–oxygen mixtures, mixtures containing iron and carbon nanoparticles and the same mixtures radiated by laser impulse. Temperature dependencies of ignition delay times in stoichiometric 20%(CH\textsubscript{4} + O\textsubscript{2}) + Ar mixture in presence of 0.3–2.0 ppm of carbon and iron nanoparticles at \(T = 1300–1900\) K and \(P = 4–6\) bar are presented in figures 6 and 7. Experimental data is compared with the experimental and modeling data for non-admixtured
Figure 6. Temperature dependence of ignition delay time in stoichiometric 20%(CH\textsubscript{4} + O\textsubscript{2}) + Ar mixture containing carbon nanoparticles: 1—pure mixture, experiment; 2—pure mixture, modeling; 3—mixture containing 0.8–1.0 ppm carbon nanoparticles; 4—0.4–0.5 ppm carbon nanoparticles; 5—approximation.

Figure 7. Temperature dependence of ignition delay time in stoichiometric 20%(CH\textsubscript{4} + O\textsubscript{2}) + Ar mixture: 1—non-admixed, experiment; 2—non-admixed, modeling; 3—containing 0.8–2.0 ppm iron nanoparticles; 4—containing 0.3–0.5 ppm iron nanoparticles; 5—approximation.

mixture at same temperatures and pressures. As one can see, a weak promotion effect was observed for carbon particles and noticeably stronger—for iron particles which could be the evidence for their catalytic activity. Values of ignition delay time observed in stoichiometric mixture of 20%(CH\textsubscript{4} + O\textsubscript{2}) + Ar containing carbon nanoparticles heated by a Nd:YAG laser
Figure 8. Temperature dependence of ignition delay time for stoichiometric 20%(CH$_4$ + O$_2$) + Ar mixture containing carbon nanoparticles heated by Nd:YAG laser impulse: 1—pure mixture, experiment; 2—pure mixture, modeling; 3—mixture containing 0.8–1.0 ppm carbon nanoparticles, no heating; 4—0.4–0.5 ppm carbon nanoparticles, no heating; 5—no heating, approximation; 6—heated carbon nanoparticles synthesized in 1%C$_6$H$_6$ + Ar at $T_5 = 2190$ K; 7—1%C$_6$H$_6$ + Ar, $T_5 = 2030$ K; 8—2%C$_6$H$_6$ + Ar, $T_5 = 1816–1881$ K.

Figure 9 and 10 show temperature dependencies of ignition delay times in stoichiometric mixture of 20%(CH$_4$ + O$_2$) + Ar containing iron nanoparticles heated by Nd:YAG laser impulse. Figure 9 presents data obtained in experiments with the same heating energy fluence 0.8 J/cm$^2$ for particles synthesized at various temperatures. One can see that the particles synthesis conditions and, thus, variations of their size in the range 10–20 nm have no significant influence on ignition delay times which are considerably shorter than ones observed without nanoparticles laser heating at temperatures below 1400 K.

Figure 10 represents data obtained with various laser radiation fluences in the range 0.2–0.8 J/cm$^2$. One can see that there are no significant differences between figure 9 and figure 10; thus, changing of energy density in that range does not affect ignition delay time. Maximum temperature of laser heated nanoparticles was measured using two-color pyrometry method at maximum of LII signals registered through the side optical windows of shock tube after the heating 0.8 J/cm$^2$ laser impulse. Temperature remains constant 3500–3900 K for carbon and 2800–3000 K for iron nanoparticles at energy fluences 0.2–0.8 J/cm$^2$ due to their evaporation, thus one can assume that active radical formation rate also remains constant.

4. Discussion
A possible influence of evaporated iron atoms on ignition kinetics was analyzed. Reaction Fe + O$_2$ → FeO + O could be one of the possible channels of active radicals formation; its rate estimations were given in [19]. Kinetic modeling had shown, though, that decreasing of ignition delay time observed in experiments could only be the result of iron atoms concentration of at least
Figure 9. Temperature dependence of ignition delay time for stoichiometric mixture of 20%(CH$_4$ + O$_2$) + Ar containing iron nanoparticles heated by Nd:YAG laser impulse: 1—pure mixture, experiment; 2—pure mixture, modeling, 3—mixture containing 0.8–2 ppm iron nanoparticles, no heating; 4—0.3-0.5 ppm iron nanoparticles, no heating; 5—no heating, approximation; 6—heated iron nanoparticles synthesized at $T_2 = 729$ K, $T_5 = 1268$ K; 7—$T_2 = 972$ K, $T_5 = 1832$ K; 8—$T_2 = 822$ K, $T_5 = 1483$ K.

1000 ppm. Moreover, consideration of the much faster compete reaction Fe + O$_2$ ↔ FeO$_2$ [20] increases the necessary iron concentration up to 0.2–0.5%, which is comparable with the total iron concentration in mixture N1. Due to low actual concentration of iron nanoparticles and limited volume where their evaporation take place considered promotion mechanism is quite improbable. Therefore it was assumed that observed promoting effect is mainly caused by catalytic reactions of methane decomposition on the surface of iron particles and subsequent initiation of combustion chain reactions by forming CH$_3$ radicals. Catalytic activity of iron was reported in [21].

For the estimation of the rate constants of such reactions, the kinetic mechanism was supplied with a first order reaction of methane decomposition

$$\text{CH}_4 \rightarrow \text{CH}_3 + \text{H} \quad (1)$$

formally describing the formation of CH$_3$ radicals in the catalytic reactions on particle surface. Modeling induction times were evaluated for various rate constants of reaction (1) and compared with the experimental data. It was observed that the values $k_1 = 30$ s$^{-1}$ and $k_2 = 6.5$ s$^{-1}$ describe the results obtained for heated and non-heated particles. Ratio $k_1/k_2$ allows estimating characteristic activation energy and pre-exponent factor of surface catalytic reaction if effective surface temperature would be provided.

In the first variant of analysis it was assumed that an effectiveness of energy transfer in collision of gas molecule with an overheated particles depends on accommodation coefficient which could be determined as a relation involving gas temperature, particle temperature and equilibrium temperature of molecules after the collision:

$$\alpha = \frac{T_{\text{mol}} - T_{\text{gas}}}{T_{\text{part}} - T_{\text{gas}}} \quad (2)$$
Figure 10. Temperature dependence of ignition delay time for stoichiometric mixture of 20%\((\text{CH}_4 + \text{O}_2) + \text{Ar}\) containing iron nanoparticles heated by Nd:YAG laser impulse: 1—pure mixture, experiment; 2—pure mixture, modeling; 3—mixture containing 0.8–2.0 ppm iron nanoparticles, no heating; 4—0.3–0.5 ppm iron nanoparticles, no heating; 5—no heating, approximation; 6–10—iron nanoparticles heated by laser impulse with various energy fluence, J/cm\(^2\).

As one can see from figures 8 and 10, characteristic temperatures in conditions of present work could be assumed to be \(T_{\text{gas}} = 1350\) K, \(T_{\text{part}} = 3300\) K. Value \(\alpha\) may vary significantly for particular gases; generally it decreases for lighter molecules and increases for polyatomic ones. There are some estimations of \(\alpha\) for helium and argon, but no estimations were given for methane. Value 0.1 was chosen for the calculations, providing \(T_{\text{mol}} = 1550\) K. Then

\[
\frac{k_1}{k_2} = \frac{e^{-E_a/RT_{\text{mol}}}}{e^{-E_a/RT_{\text{gas}}}},
\]

and

\[
\frac{E_a}{R} = \frac{\ln (k_1/k_2)}{1/T_{\text{gas}} - 1/T_{\text{mol}}},
\]

and \(E_a = 132\) kJ/mol, i.e. substantially lower than activation energy of same reaction in gas phase (380–440 kJ/mol according to various sources). Pre-exponent \(A\) of (1) thus can be estimated as \(A = 8.5 \times 10^5\) s\(^{-1}\). One should note that experimentally found volume fraction of iron nanoparticles 1 ppm and average size 10 nm one could estimate the particle concentration as \(10^{12}\) cm\(^{-3}\) and the methane-particle collision rate as \(1.5 \times 10^5\) s\(^{-1}\). Thus obtained \(A\) value could be considered to be in accordance with gas-kinetic collision frequency.

Another consideration assumes that during collision of methane molecules with the particles some kind of activated complex could be formed which exists for a time much longer than is necessary for equilibration of internal energy of methane molecule with the temperature of the particle. In this case \(T_{\text{mol}} = T_{\text{part}}\) and \(E_a = 19\) kJ/mol, while much less value of pre-exponent \(A = 35\) s\(^{-1}\) reflects the low probability of formation of such a complex.
5. Conclusion

Notable promoting influence of iron heated nanoparticles on the ignition of diluted stoichiometric methane–oxygen mixture was observed experimentally. Presence of 0.3–2.0 ppm of iron heated nanoparticles decreases ignition delays twice at the temperatures below 1400 K, while addition of 0.4–1.0 ppm of carbon nanoparticles did not result in noticeable promotion. Impulse laser heating of iron nanoparticles sizes 10–20 nm at 0.5–1.0 ppm concentration suspended in stoichiometric methane–oxygen mixture with 0.13–0.80 J/cm² fluence resulted in notable shortening of ignition delays. Smaller effect was registered at laser heating of carbon nanoparticles sizes 20–40 nm at similar concentrations and laser intensities. Iron nanoparticles supposedly increase active radical concentration due to catalytic surface reactions of CH₄ and O₂ decomposition and thus initiate chain combustion reactions. The estimations of effective activation energy and probability of such a catalytic reaction were given in two various assumptions.

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