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PHOTOCHEMICAL IGNITION STUDIES. III. IGNITION BY EFFICIENT AND RESONANT MULTIPHOTON PHOTOCHEMICAL FORMATION OF MICROPLASMAS

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This is the third of a series of reports concerning the activation (ignition) of reactive gases using focused ultraviolet lasers. The goal of this research is to ascertain the potential of uv laser multiphoton photochemical ignition as a primary igniter or ignition augmentation source for propellants or their pyrolysis products. In this report, ignition properties of premixed H_2/O_2 and H_2/N_2O flows at atmospheric pressure have been studied. Tuning the laser in the 225.6 nm wavelength region has yielded three minima in the amount of incident laser energy (ILE) that is required to ignite either mixture. The minima correspond exactly to the two-photon resonant excitation wavelengths for the three spin-orbit split ground electronic states of oxygen atoms. A determination of the ILE necessary to ignite both premixed flows as a function of equivalence ratio shows a minimum far into the fuel-lean region. Also, the minimum ILE value for the ignition of H_2/O_2 was found to be around 0.3 mJ, while ignition of the same mixture with the green beam from a frequency doubled Nd:YAG laser (532 nm) required an ILE value near 13 mJ. Additional time-resolved

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spectral studies were carried out on O_2 and N_2O flows alone. These indicated a resonant formation of a microplasma with a lifetime on the order of 100 nsec. All of these results lead to the conclusion that multiphoton photochemical ignition is a phenomenon consisting of three major components: (1) the multiphoton photochemical formation of oxygen atoms; (2) multiphoton ionization of these atoms to efficiently form free electrons in the laser focal volume; (3) the formation of a laser microplasma using the electrons formed in the previous process as seed electrons. As such, this new laser ignition source appears to be more efficient and more controllable than the well-known laser-produced spark (gas breakdown) process and it thus should be useful for further ignition studies.

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I. INTRODUCTION

There has been a growing interest recently in the application of lasers which operate in the ultraviolet region to the ignition of reactive mixtures. One such study has described the use of the excimer lasers F_2 (157 nm) and ArF (193 nm) for the ignition of H_2/O_2 or H_2/air mixtures by photolysis of O_2 into O atoms.[1] More recently, experiments have been undertaken in which a KrF (248 nm) excimer laser has been used to photolyze O_3 into $O + O_2$ in the presence of CH_4 and H_2/O_2.[2] Both of these experiments have involved single photon photochemistry. Ignition of reactive gas mixtures which used multiphoton photochemistry was first demonstrated in our laboratory on simple hydrocarbon/air or N_2O mixtures using the ArF and KrF excimer lasers.[3] Interpretation of those results was based to a large extent on related experimental work in our and other laboratories which showed that uv laser (particularly the ArF excimer) multiphoton interaction with small carbon-containing fuels can be very extensive and can lead to substantial photofragmentation and fragment excitation. A particularly illustrative case involves the C_2H_2 molecule which upon irradiation by the ArF laser yields ground and excited state radicals such as C_2H, C_2, CH,[4] as well as the H and C atoms and C^+ ions.[5] On the basis of these results, it was not surprising to find that a C_2H_2/air mixture required only 0.25 mJ of incident laser radiation to ignite.

In a recent preliminary study we have observed a strong wavelength dependence in the amount of incident laser energy required to ignite a H_2/O_2 flowing mixture using a tunable laser system near 225.6 nm.[6] Specifically, we found that the most efficient ignition wavelength corresponded to the peak of the two-photon resonance excitation process for oxygen atoms in the J=2 ground spin-orbit state. The focused ultraviolet laser not only apparently caused photodissociation of O_2 into O atoms, but also, when on O-atom resonance, required the least amount of energy to ignite the gases. Furthermore, a plot of incident laser energy as a function of equivalence ratio yielded a minimum at 0.61, far from stoichiometry. This result further reinforced the conclusion that the laser-oxidizer interaction is an important element in the ignition process of H_2/O_2 mixtures at 225.6 nm.

These preliminary experiments, however, were not detailed enough to identify the specific mechanism(s) involved in the ignition process. In particular, laser two-photon resonant population of the oxygen atom $3p\ ^3P$ states at 88,630 cm^{-1} (10.99 e.v.) can lead to a number of processes including excited state chemistry, heat deposition at the focal volume due to quenching collisions, and/or the absorption of a third photon leading to the formation of O^+ ions and free electrons.[7] It is the purpose of this report to describe an experimental effort aimed at a much more comprehensive characterization of the multiphoton photochemical ignition phenomenon. The results presented here indicate that the ion formation channel is a key process since it represents an efficient and direct route for the production of the initial free electrons early enough in the laser pulse such that they become the seed material for the creation of a laser-produced spark, i.e., microplasma. The primary role of this short-lived microplasma (ca. 100 nsec) apparently is to be a localized source of highly reactive chemical intermediates at a very high temperature. If the spark is intense enough, then the resultant ignition kernel is sufficiently strong to permit transition into full combustion.

II. EXPERIMENTAL

The experimental schematic is given in Figure 1. Since it has been described in detail previously,[6,8] only the major points will be highlighted. Tunable uv laser radiation in the 225.6 nm region was focused with a 50 mm focal length lens at a position 1-2 mm above the burner surface. Typical laser energies up to 1 mJ/pulse yielded power densities around 10^{11} W/cm^2 in the focal volume. The water-jacketed H_2/O_2 burner was fabricated from a stainless steel Swagelock 0.25 in. terminator fitting through which a 0.9 mm hole was drilled. Matheson (Model 620) flowmeters were calibrated by a GCA Precision Scientific wet test meter for H_2, O_2, and N_2O flows up to 2 LPM. This resulted in orifice linear flow velocities in the 10^3 cm/sec range. The incident laser energies were always measured just before the focusing lens with a Scientech (Model 38-0103) disc calorimeter-power/energy meter. The emission signals were detected, averaged, and processed as described previously.[8] The excitation wavelength scans were performed manually, and each emission wavelength data point represented the average value for 512 laser shots.

Time-resolved emissions were digitized with a Tektronix 7912AD digitizer (7A24 amplifier and 7B90P timebase) and accumulated in a PDP-11/04 computer. The response time is ca. 25 nsec FWHM (see Figure 6) due to the relatively slow response of the EMI 9558QA photomultiplier detector tube. The power dependence of the O-atom emission intensities at 777.5 nm for O_2 and N_2O flows was measured using a 200 mm focal length lens to avoid the formation of microplasmas and only to measure the photon dependence for the photolysis of those two molecules. For these experiments, the Nd:YAG laser amplifier flashlamp energy was varied, as before,[8] to change the output power at 225.6 nm.

III. RESULTS AND DISCUSSION

A. Ignition

Figure 2 shows the wavelength dependence of the amount of incident laser energy necessary to ignite a premixed flow of H_2/N_2O. A similar type of behavior has been found for H_2/O_2 premixed flows. The curve clearly shows a strong dependence of the incident laser energy (ILE) on the laser wavelength with three prominent features around 225.6, 226.0, and 226.2 nm. The wavelengths of these three features are exactly the same as the fluorescence peaks which result from oxygen atom two-photon excitation of the ground electronic spin orbit split states J=2, J=1, and J=0, respectively.[7-8] This result unequivocally indicates that the electronic excitation of oxygen atoms is an important feature of the ignition mechanism. Also, the spectral widths of these features are considerably broader than those observed during the flame O-atom excitation scans. The reason for this difference will be discussed in the next section.

The dependence of the ILE on the equivalence ratio for H_2/O_2 flows is given in Figure 3. The lower trace results from the laser wavelength set at the peak of the O-atom two-photon excitation, while the upper trace is for the green laser beam, i.e., the second harmonic of the Nd:YAG laser (532 nm). Two points relevant to this figure should be discussed. The first is the

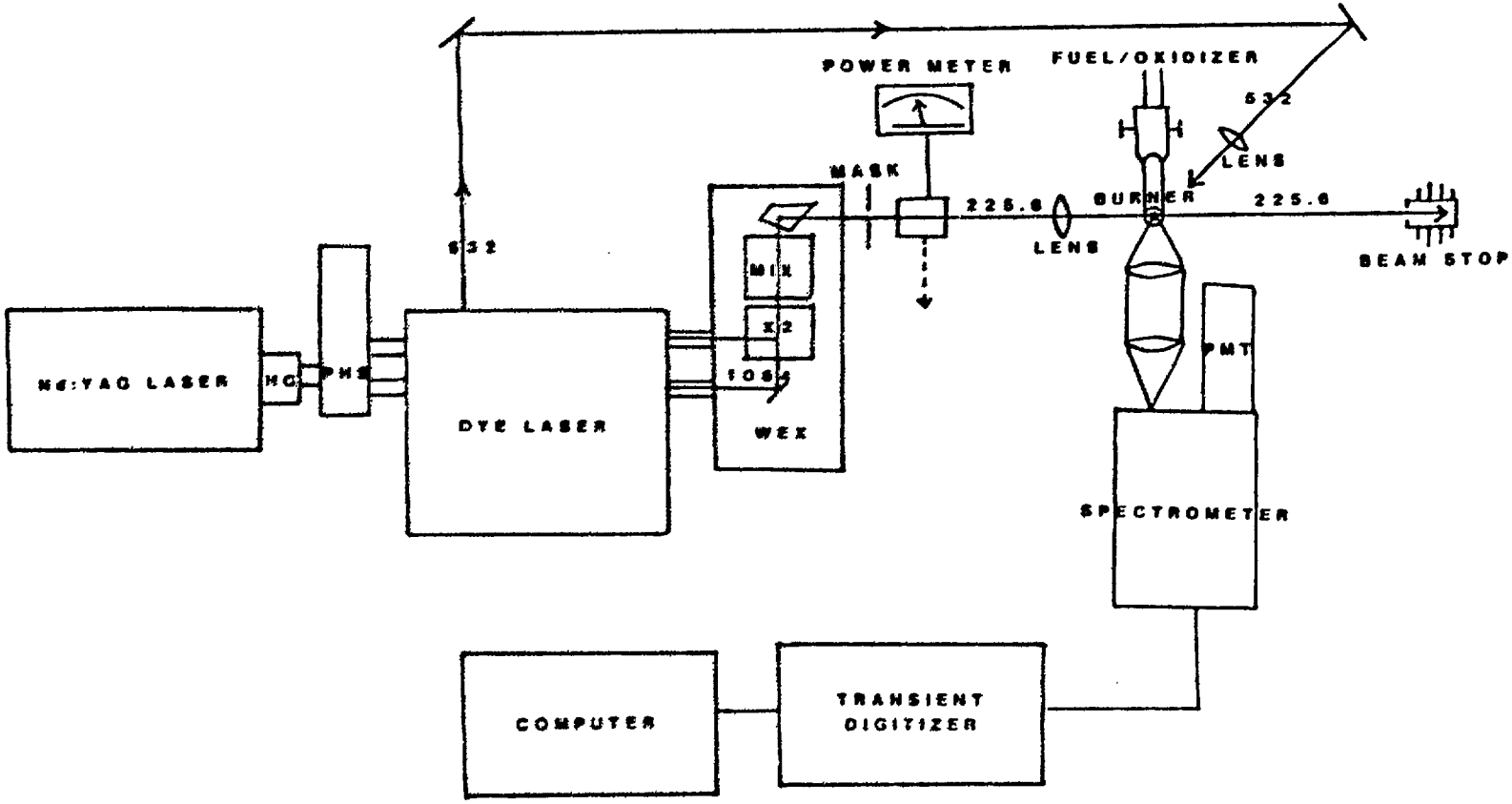

Figure 1. Experimental Schematic

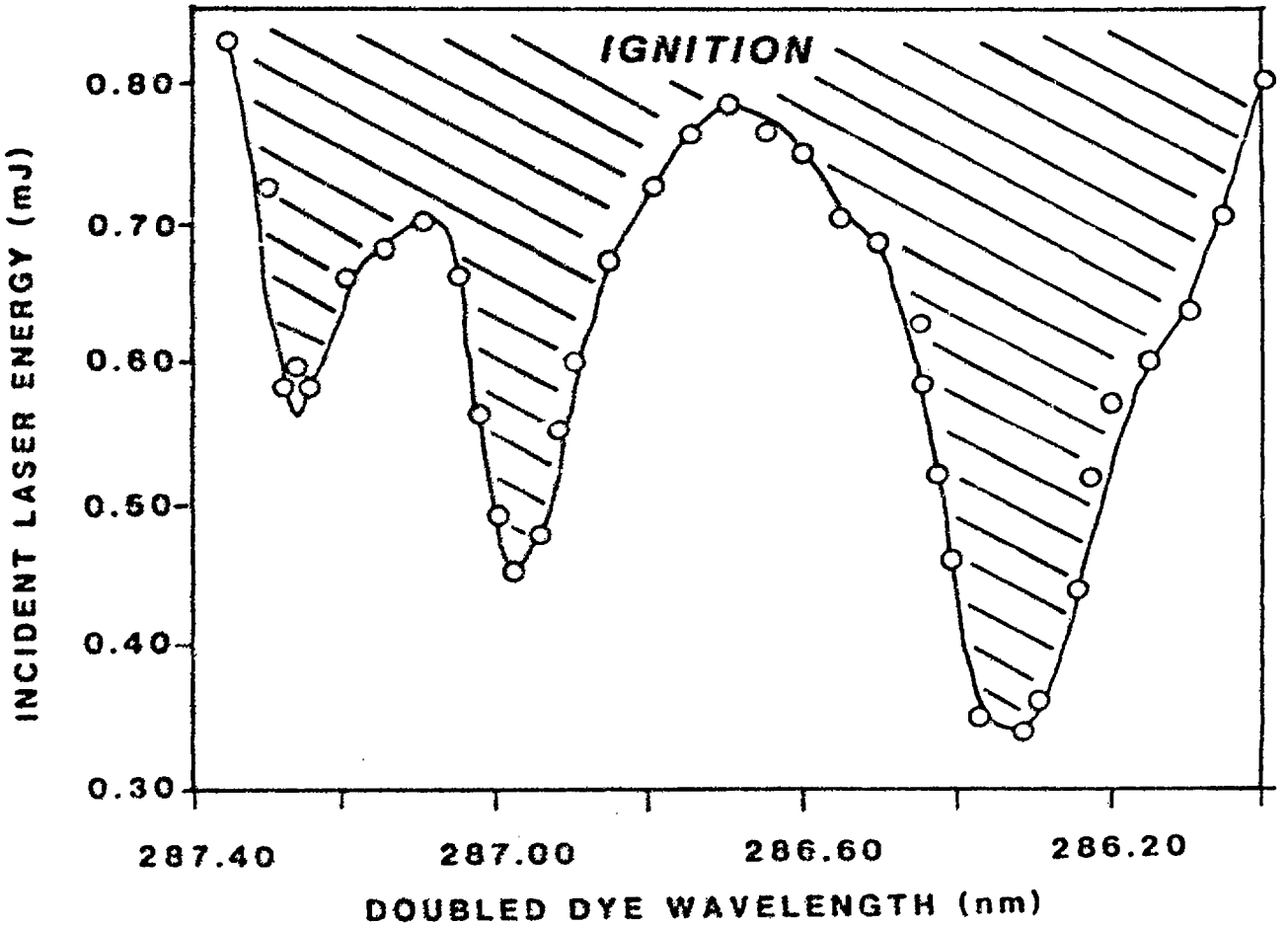

Figure 2. Incident Laser Energy Necessary to Ignite a Premixed Flow of H_2/N_2O as a Function of Laser Wavelength in the 225.6 nm (Doubled Dye +1.06 Micron) Region

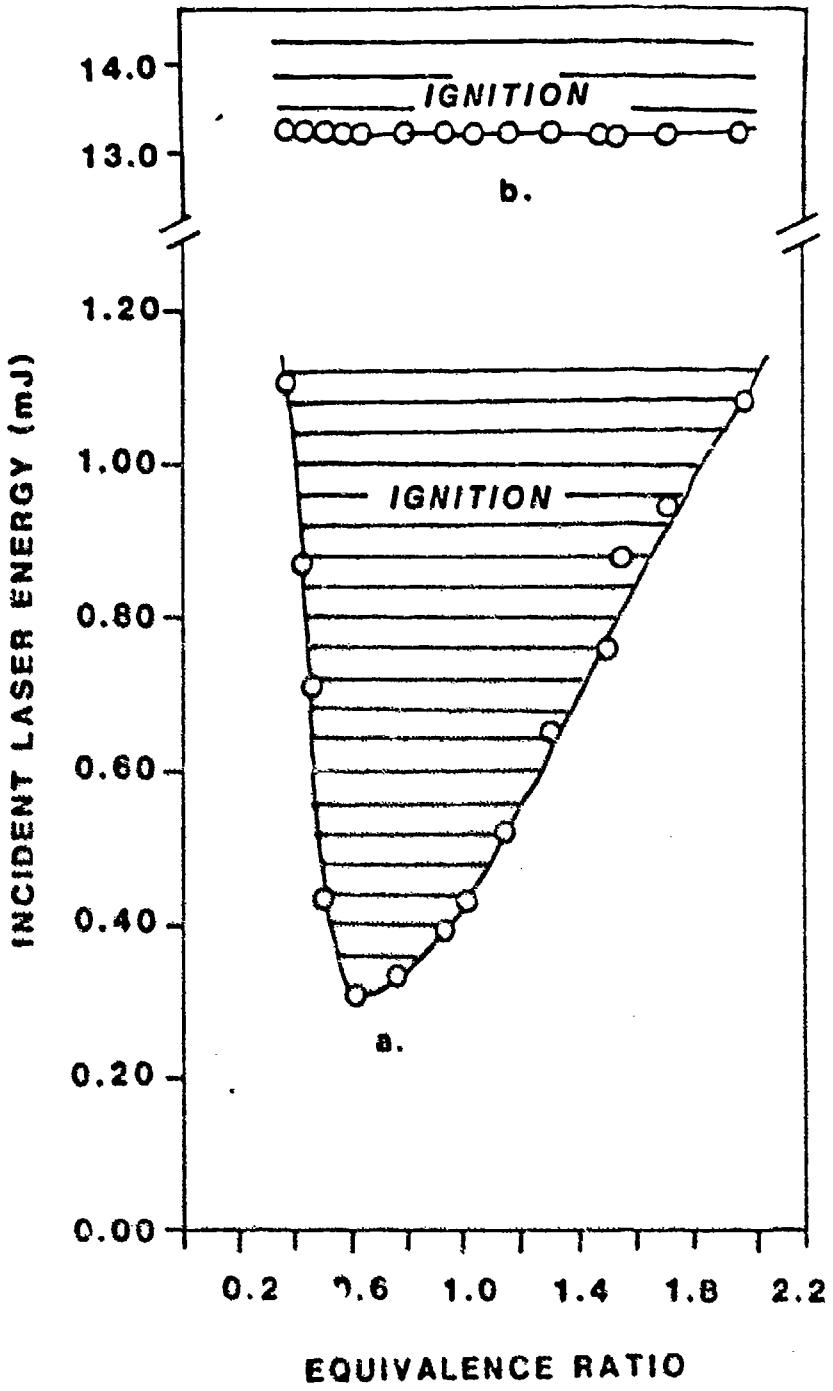

Figure 3. Dependence of the Incident Laser Energy Required to Ignite a H_2/O_2 Mixture as a Function of Equivalence Ratio (a) For the Laser Wavelength Set at the Peak of the O-Atom Two-Photon Excitation, (b) For the Nd:YAG Second Harmonic (532 nm)

observation that the minimum in the lower trace is far into the fuel-lean region. A similar type of behavior was noted for H_2/N_2O flows. The reason for this appears to be the same as before, i.e., the uv laser is clearly interacting significantly with the oxidizer (O_2) component of the reactive flow. Observation of the minimum so far from the stoichiometric point is, of course, in sharp contrast with the usual behavior found for spark ignition in a closed bomb. However, the recent report on the excimer laser ignition of H_2/O_2 mixtures also noted the most efficient ignition to be in the fuel-lean region.[1]

The second point relates to the upper trace of Figure 3 where ignition was caused by the green laser. Not only is the actual value of the ILE much higher at 532 nm than at 225.6 nm, but also the 532 nm dependence is virtually flat across a very wide range of equivalence ratios. This type of behavior has been observed previously in our initial ignition studies involving hydrocarbons[3] and can be explained by the inherent properties of the laser "spark" (gas breakdown) process. Specifically, due to the sharp threshold associated with the onset of absorption of laser energy, the spark (plasma) when produced, is typically much more energetic than the required critical ignition energy.[9] Furthermore, there is usually a sizable blast wave associated with the spark.[9] As indicated in Figure 3b, the spark intensity is sufficient to ignite mixtures at either extreme of equivalence ratios, and is clearly much greater than necessary for near stoichiometric mixtures.

Implicit in our discussions of multiphoton photochemical ignition of H_2/O_2 and H_2/N_2O is the fact that the process first has to start with the photoproduction of the oxygen atoms in the ground ^3P state. In order to study this process, we measured the laser power dependence for the production of the two-photon excited oxygen atoms whose fluorescence was detected at 777.5 nm.[8] The reason for doing this is that frequently such a power dependence study will indicate how many photons are involved in the process. Experiments were undertaken on flows of O_2 and N_2O respectively using a 200 mm focal length lens to avoid problems of microplasma formation. These measurements indicated that the photochemical formation of ground state oxygen atoms was a multiphoton process for both O_2 and N_2O requiring two photons in each case, i.e., for both cases we measured a four photon dependence for the O-atom emission. This is consistent with our previous measurement for H_2O,[8] whereas for O_2 this quantity had not been measured previously. The implication of these findings to our ignition studies will be discussed in the next section.

B. Microplasma Formation

During the course of our ignition experiments we began to take note of a faint source of white light that emanated from the laser focal volume region. The intensity of this light was clearly wavelength dependent with the brightest emission occuring at the wavelengths corresponding to the peaks of the O-atom two-photon excitation. In order to study this behavior in greater detail, we initiated both spectral and temporal studies of these microplasmas for O_2 and N_2O flows using the 50 mm focal length lens.

Figure 4a shows the excitation curve for O atom emission at 777.5 nm where the O atoms were themselves generated by the same laser focused into the O_2 flow. A similar plot (Figure 4b) is also included for nascent O atom two-photon excitation in a stoichiometric H_2/O_2 flame. Furthermore, two ignition

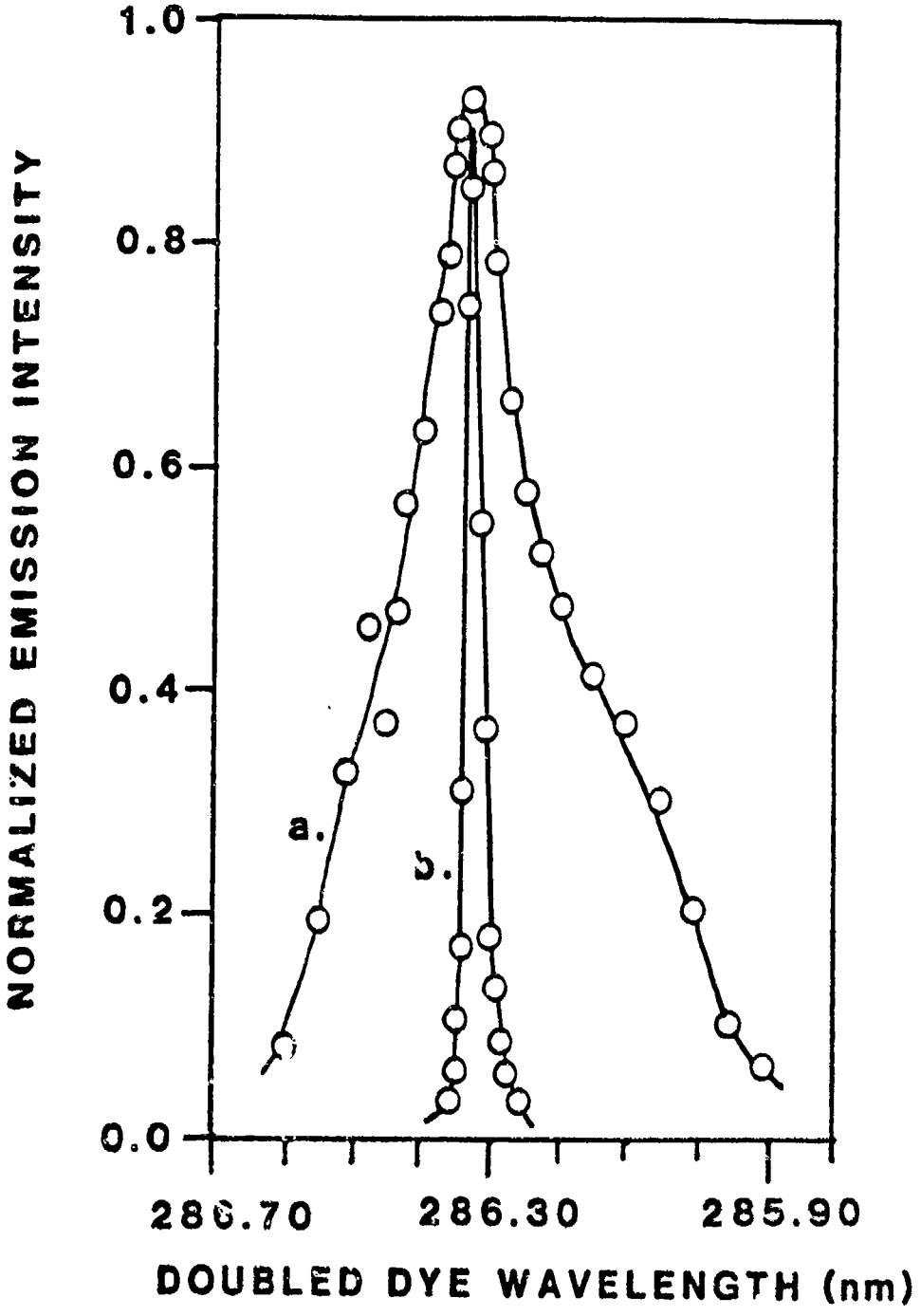

Figure 4. Excitation Curve for O-Atom Emission at 777.5 nm from (a) an O_2 Flow, (b) a Stoichiometric H_2/O_2 Flame

plots for fuel-lean and stoichiometric H_2/O_2 mixtures are given in Figures 5a and b, respectively, with the differences in the absolute value of the ILE explained previously.[6] A number of similarities and differences can be seen and discussed. In particular, the spectral width for O-atoms produced in a O_2 flow is much greater than for the nascent flame O-atoms. The substantial spectral width in the O_2 flow is, however, quite comparable to the ignition profile in the fuel-lean case, while the spectral profile for the ignition of a stoichiometric mixture is somewhat narrower and less efficient than the fuel-lean case. The explanation for these observations is that the spectral profile in the ignition case as well as in the case of the O_2 flow, is not really a representation of an atomic spectral property, but rather an indication of a much more complex process, i.e., the formation of a microplasma, which is inherently a highly nonlinear phenomenon. It is well-established that the laser-produced microplasma (spark) needs seed electrons in order to grow. In our case it finds them in the spectral wings of the two-photon resonant, three-photon ionization of oxygen atoms. When these free electrons are formed in the early part of the laser pulse, then the cascade plasma formation mechanism is initiated and the plasma is ultimately heated up to a very high temperature by the inverse brehmsstrahlung effect.[10-12] Thus, when this occurs, it is no longer valid to consider the O-atom emission at 777.5 nm as a simple two-photon laser induced fluorescence process. It is therefore not surprising that the spectral behavior of the ignition of premixed gases, a process sensitive to microplasma formation, should be similar to that of the microplasma producing precursor alone. This is, in fact, what is observed for the O_2 flow and the fuel-lean reactive mixture in Figures 4a and 5a. The fact that the stoichiometric ignition curve shows a narrower width and is less efficient as well, is explained by the fact that the H_2 hampers the growth of the microplasma, presumably due to its high ionization potential (I.P. for O_2 = 12.063 e.v. and for H_2 = 15.427 e.v.), and thus the plasma is relatively less intense. In search for other explanations for these wide spectral widths, a wavelength dependence of the initial step, i.e., multiphoton photochemical production of atoms, should be considered. It would be most unlikely, however, for such a dependence to yield the similar type of spectral profile which is found for all three spin-orbit components (Figure 2).

An additional parameter that was investigated is the temporal behavior of the O-atom emission at 777.5 nm. Figure 6 shows the time-resolved emission for scattered laser light (Figure 6a), flame O-atoms (Figure 6b), and O-atoms produced in the O_2 flow under conditions of microplasma formation (Figure 6c). Clearly the lifetime is much longer for the O_2 flow case since it actually is related to the lifetime of the plasma with direct laser produced signals from simple multiphoton photolysis and the two-photon excitation being a factor only in the leading edge of the trace.

The role of excited state O-atom chemistry has not been explicitly considered so far. The fact that a microplasma exists in multiphoton photochemical ignition implies that laser-populated excited state chemistry is probably not important since the kinetics of the microplasma process would appear to overtake other competing processes. Furthermore, we undertook a series of ignition studies of H_2/O_2 flows using longer focal length lenses (100 mm, 150 mm, 200 mm) and found that the ILE required to ignite increased considerably and ignition did not occur without the formation of the microplasmas. On the other hand, even though our experiments strongly

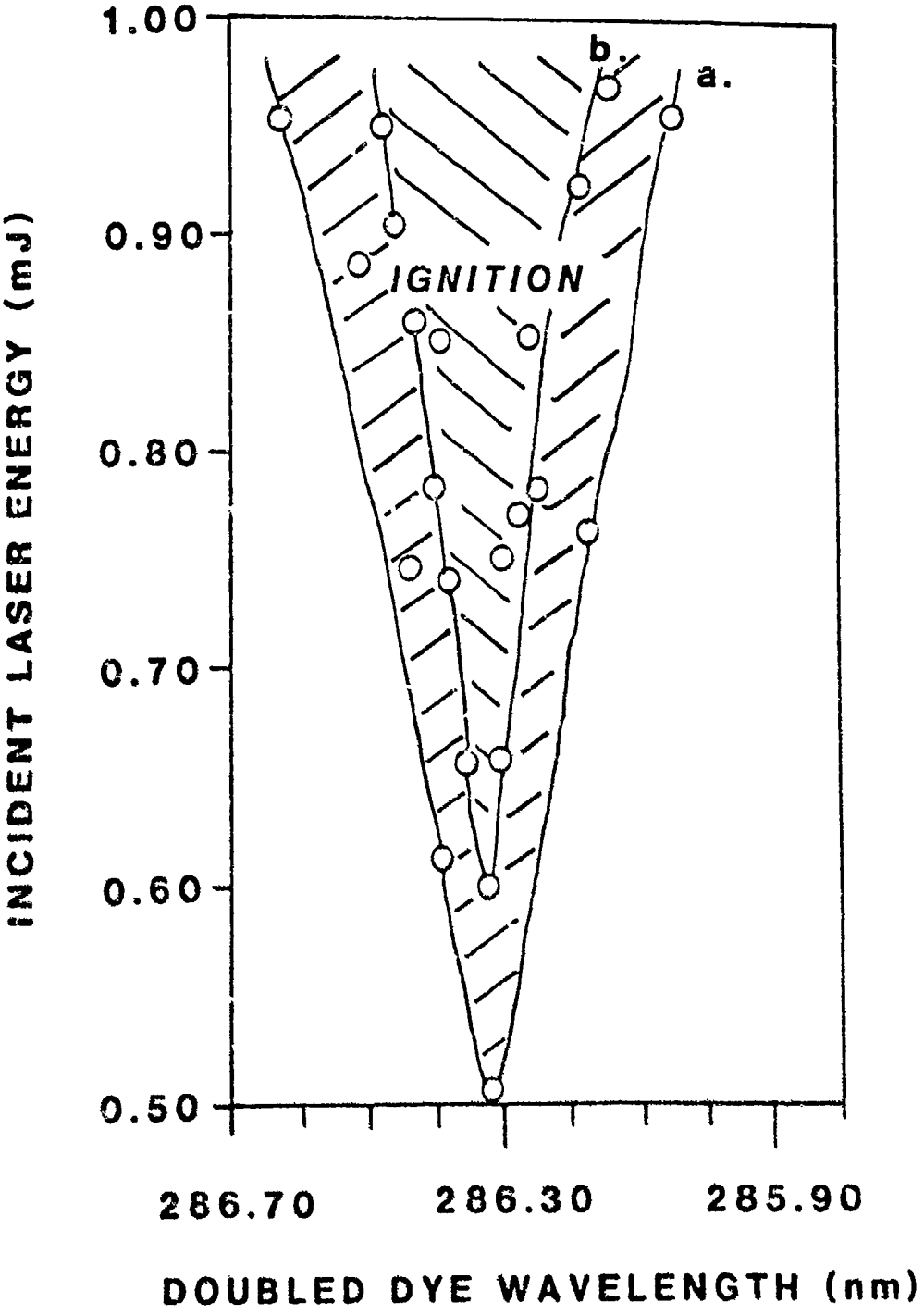

Figure 5. Incident Laser Energy Necessary to Ignite a Premixed Flow of H_2/O_2 Which is (a) Fuel-Lean (Equivalence Ratio = 0.6) and (b) Stoichiometric (Equivalence Ratio = 1)

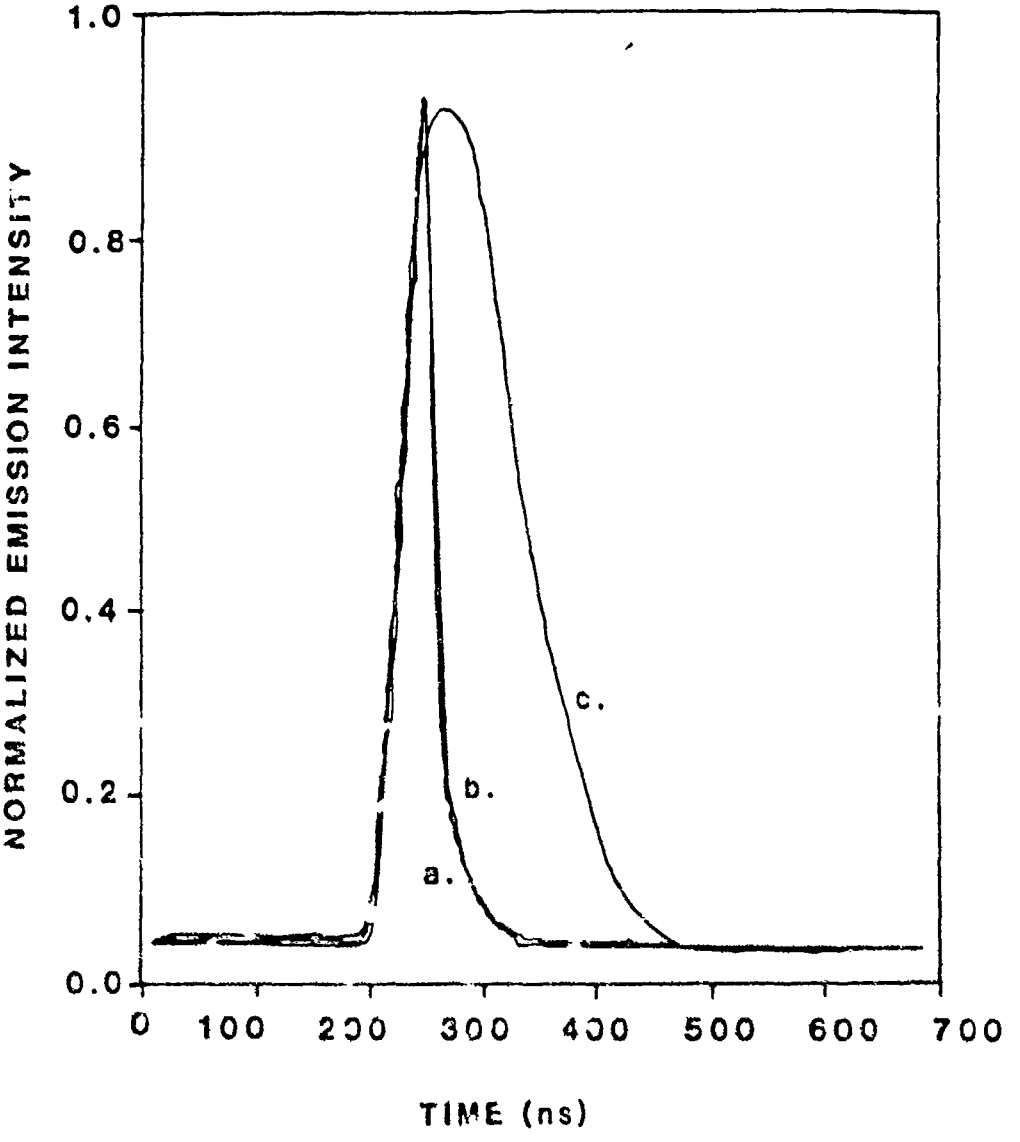

Figure 6. Time-Resolved Emission From (a) Scattered Laser Light, (b) Flame O-Atom Emission at 777.5 nm, (c) O-Atom Emission at 777.5 nm from Microplasmas Formed in O_2 Flows

implicate the microplasma formation process as a key element in ignition, we cannot discount the possible importance of the photochemical formation of radicals in the converging laser beam near the microplasma. These radicals could very well be important in the early stages of ignition kernel growth, but after the microplasma had decayed. Clearly, future experiments with high speed photography, as well as in a closed bomb and with other appropriate optical diagnostic tools, would be very helpful in promoting further understanding of this phenomenon.

IV. CONCLUSION

Focused ultraviolet laser radiation is capable of activating reactive gas mixtures through a new, previously unreported mechanism involving multiphoton photochemistry, ionization, and microplasma formation. The major difference between this work and previous work on laser spark formation is that multiphoton photochemical ignition provides a more efficient and controllable means for liberating the free electrons which then lead to the laser spark formation process. Due to these virtues, this laser ignition phenomenon should open up new opportunities in ignition studies. Also, since lasers possess certain attractive characteristics such as beam propagation through great distances, as well as excellent time-resolution, there may be new opportunities for practical applications which require the activation of reactive systems.

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