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6. AUTHOR(S)
Lisa Pfefferle and Charles McEnally

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Yale University
New Haven CT 06520

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14. ABSTRACT

An excimer laser was purchased to perform in-situ measurements of acetylene in fuel-rich flames. The acetylene measurements were performed by fragmenting acetylene with the intense 193 nm wavelength laser output and then detecting the fluorescence from CH and C2 fragments that were formed in excited states. The measurements were compared with mass spectrometer measurements and were shown to agree spatially. The data were used to determine where a soot model accurately predicted the spatial distribution of hydrocarbon growth regions in flames and the trends followed by the peak acetylene concentrations in response to changes in combustion conditions.

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FINAL REPORT
FY00 DURIP
Grant # F49620-00-1-0214

Professor Lisa D. Pfefferle
Yale University
Department of Chemical Engineering

THE PROPOSAL: **Excimer Laser Fragmentation-Fluorescence Measurements of Acetylene to Advance Fundamental Understanding of Soot Formation**

TOTAL FUNDS REQUESTED FROM DOD: $116, 900

DATES: 31-03-00 to 31-03-01

Excimer Laser Fragmentation-Fluorescence Measurements of Acetylene to Advance Fundamental Understanding of Soot Formation

FY00 DURIP

Final Report AFOSR Equipment Grant
Grant # F49620-00-1-0214

Lisa Pfefferle and Charles McEnally, Yale University

ABSTRACT

We are using the new excimer laser purchased on this grant to perform in-situ measurements of acetylene in rich flames. Future studies will also include generation of tunable VUV light for use in a single-photon photoionization mass spectrometer. The acetylene measurements are performed by fragmenting the acetylene with the intense 193 nm laser output and then detecting fluorescence from CH and C_2 fragments that are formed in excited states. These in-situ measurements have been compared with MS measurements and shown to agree spatially. This data was used to improve kinetic mechanism input to a soot model being developed with DOD funding. The data determined where the model accurately predicts the spatial distribution of hydrocarbon growth regions in the flame and the trends followed by the peak acetylene concentrations in response to changes in combustion conditions, allowing model refinement. This paper will be submitted to the next International Combustion Institute meeting. The in-situ measurements have confirmed that the sample probe results are not affected by reactions inside the probe. The acetylene diagnostic will also be suitable for imaging of acetylene concentrations in complex, unsteady flows.

In future studies, the tunable VUV light made possible using the excimer laser will add isomer-selectivity to the mass spectrometer. This capability will be used to distinguish between various proposed aromatic hydrocarbon formation and growth pathways in experiments where small concentrations of hydrocarbons are added to the fuel in well-characterized methane flames.

The report is divided in two parts the Part 1, the Technical Section describing motivation, set-up, use of the laser and preliminary results and Part 2: the Financial section.

Part 1: Technical Section

1 Background

1.1 Adverse Impacts of Soot Formation in Military Aircraft

The production of soot particles in aircraft engines creates both general environmental problems and operational difficulties, which provides a strong motivation to understand and limit the chemical mechanisms responsible for particulate formation.

From the environmental standpoint, aircraft contribute to the atmospheric aerosol levels via high altitude soot emissions. These aerosols influence such important problems as global climate change and ozone depletion by affecting the atmosphere's radiation balance, cloud and contrail formation, and upper troposphere water vapor removal. Furthermore, increasing evidence indicates that human exposure to fine particles, such as soot, leads to tens of thousands of deaths per year in the United States. This evidence led the US Environmental Protections Agency to propose new ambient air quality standards for fine particles last summer [Kaiser, 1997].

In terms of operational difficulties, high soot concentrations in combustor primary zones contribute to high thermal radiation loads on combustor liners, which are of concern because ceramic thermal barrier coatings are usually partially transmissive in the infrared. Soot coatings on liner surfaces will drastically increase underlying metal temperatures. Similar concerns arise from radiation loading of turbine blades. For military engine design, there are concerns about soot emissions serving as contrail formation sites, thus enhancing the tactical visibility of aircraft. Further, impaction of soot particles on radar-reflecting low observable surfaces can compromise the radar signature of an aircraft.

2 Results

2.1 Laser Purchase and Set-up Summary of Activities

The requested laser, a Complex 205 Excimer Laser Multigas with (Halogen) Fluorine Source, was ordered on March 28, 2000 per Yale Purchase Order P.O. YKP1037684. The financial section of this report (Part 2) lists the expenditures on this grant along with the cost shared component.

The laser arrived in the late summer of 2000. The laboratory had been prepared by in advance installing vibration, isolation and cooling facilities and by upgrading our data acquisition capability with a new Le Croy oscilloscope. The laser was installed and performance checked (exceeded manufacturer specs in terms of energy per pulse).

In September 2000, the optics for focusing the beam through a coflowing, nonpremixed burner were set up and the arrangement was approved by Yale's laser safety officer.

The first qualitative experiments were carried out: 193 nm excitation spectra were measured from air and at various locations in a methane flame. Air spectra contained the Schumann-Runge bands of oxygen from 200 to 300 nm, and were similar to published spectra of oxygen excited at 193 nm. Flame spectra from fuel-lean regions showing some oxygen bands, and additional hot bands due to absorption of the 193 nm photons by oxygen molecules thermally excited to excited vibrational states. Flame spectra from fuel-rich regions showed numerous bands of C-atom, CH and C_2 due to photodissociation of flame hydrocarbons into electronically excited fragments. These bands were identified and matched by comparing them with published 193 nm spectra of acetylene and other hydrocarbons in photolysis cells. The CH and C_2 bands were between 300 and 700 nm and in most cases were not subject to interferences from the oxygen bands. The spatial maxima of the C, CH and C_2 bands corresponded to the pyrolysis zone of the methane flame as determined by on-line mass spectrometry, and little if any signal was observed in the fuel-lean or unburned fuel portions of the flame.

These results show that the spectral features in the flames are similar to those observed in static cell experiments. Spatial profiles of the C_2 and especially CH bands correlated spatially with profiles of acetylene concentration measured with probe sampling/mass spectrometry. This is encouraging for use of the fragmentation-fluorescence diagnostic for in-situ measurements of acetylene and as a pyrolysis zone marker. Interference from fragmentation of other species does not measurably interfere in methane flames however this will have to be assessed on a flame by flame basis.

2.2 *AFOSR-Supported Soot Modeling at Yale University*

Many computational and experimental studies of soot formation mechanisms in laboratory-scale flames have been initiated in recent years. One of these efforts was a collaborative program that involves Yale University and the United Technologies Research Center, which is supported by AFOSR (Contract F49620-94-C-0059) with Dr. Julian M. Tishkoff as contract monitor. The principal investigators for this project were Mitchell D. Smooke (Yale) and Meredith B. Colket and Robert J. Hall (UTRC).

The immediate objective of their program was to create and refine a fundamental model of soot formation in flames. The year 2000 version of the model combines a detailed aromatic hydrocarbon and soot particle formation/growth mechanism [Hall et al., 1997] with a sophisticated flame structure model [Smooke et al., 1992] that includes mixture-specific transport properties, detailed fuel oxidation chemistry, and radiation heat transfer. Thus, in this model soot particle inception, surface growth and oxidation rates are computed from benzene, acetylene, and OH mole fractions calculated with relatively detailed flame chemistry. In most other existing soot models there rates are computed from variables such as mixture fractions that are easier to calculate, but only indirectly connected to the rate in question.

Ultimately, the goal of developing soot models is to allow engine designers to predict and limit soot levels in real devices without testing of prototypes. Currently, computational fluid dynamic simulations are useful to engine designers for predicting NOx emissions, but not soot formation [Correa, 1998]. Developing a fundamentally correct soot model that can successfully predict soot formation in a wide range of laminar flames is a necessary step towards identifying those phenomena that must be included in successful industrial-style simulations. We have made progress in this area as noted below and a joint paper with Smooke and Pfefferle at Yale and Colket and Hall at UTRC is under preparation for submission to the next combustion symposium.

2.3 Our Role in Experimentally Validating the Soot Model

Testing and refining computer models requires experimental data for comparison. Since the Smooke/Colket/Hall model includes so many fundamental phenomena (fuel oxidation chemistry, heat and mass transport, radiation from soot and gas-phase species, aromatic hydrocarbon formation and growth, soot particle inception, surface growth, coagulation, oxidation, and transport) the widest possible range of experimental data is needed, both in terms of the variables measured and the flame conditions investigated (e.g. different fuels, different levels of fuel/oxidizer premixing, different burner geometries, etc.). Since acetylene is a key input for the correlative aspects of the model in-situ backup of this measurement is essential especially (as noted below) due to the fact that the initial models did not well predict the spatial profiles measured by mass spectrometry.

In our laboratory at Yale we have been experimentally studying aromatic hydrocarbon and soot formation/growth for several years, and have developed and implemented many relevant diagnostics. These include thermocouple measurements of temperature and soot volume fraction [McEnally et al., 1997]; thermophoretic sampling/TEM analysis of soot particle size and morphology [Koylu et al., 1997]; mass spectral measurement of major combustion species and C1 to C12 hydrocarbon concentrations [McEnally and Pfefferle, 1996]; laser-induced incandescence measurements of soot volume fraction [McEnally and Pfefferle, 1999b]; and laser-induced fluorescence measurements of OH, formaldehyde, and polycyclic aromatic hydrocarbons [McEnally and Pfefferle, 1999b].

These species and soot volume fraction data have been compared with the full soot model in a burner-attached methane flame [Smooke et al., 1998] and a lifted ethylene flame [McEnally et al., 1998] and data that has been compared with the flame structure and hydrocarbon growth portions of the model in a lifted methane flame [McEnally et al., 1999] and a series of partially premixed methane flames [Bennett et al., 1999].

These comparisons indicate that the overall flames structure is well predicted, even in the presence of soot, but that improvement is needed in predicting hydrocarbon and soot production. Soot volume fractions predicted by the year 2000 model were of the correct order of magnitude but the agreement in the spatial distribution was poor. Material balance comparison with our species profiles showed that predictions of benzene and to a lesser extent that acetylene were likely the cause of the soot distribution problems. A backup measurement technique was required to confirm wither the model or MS

hat the spatial distributions of acetylene have been confirmed
mer laser from this grant we were able to identify the problem as
al model used. Conversations with Professor Hai Wang (University
d steps in the C2 through C6 reaction pathways to address and we
ng predictions from his model with our data. Qualitatively, the
predicted spatial distribution of soot in the flame is significant. This
tten up as a paper to be submitted to the coming International
Combustion Symposium in Japan.

2.4 Our Studies of Hydrocarbon Growth Mechanisms

In addition to providing data for testing and refining the soot model, we have also extensively studies mechanisms of aromatic hydrocarbon formation and growth. Formation of aromatics is a necessary and frequently rate-limiting step to soot formation, but the chemical mechanisms involved are uncertain. This is especially true for the relatively large and complex hydrocarbons that are found in aircraft engine fuels since most research on detailed chemical mechanisms has focused on simple fuels such as methane and ethylene.

Our basic approach has been to measure hydrocarbon and soot profiles in methane flames with and without small quantities of various hydrocarbons added to the fuel. By choosing additives that perturb specific hydrocarbon growth pathways, we can determine whether those pathways are important to soot formation. Furthermore, since the additive is present in small concentrations, it can be a large molecule representative of real fuels while the overall flame remains diagnostically manageable.

Some of the conclusions of these studies so far that are relevant to soot formation in aircraft engines include:

Our additive experiments are continuing, with financial support from the National Science Foundations and from the Environmental Protection Agency. One goal is to continue looking for aromatic growth pathways for compounds like those in practical fuels that may be different from the ones that have been identified for very simple fuels such as methane and acetylene. Another goal is to examine additives that reduce soot concentrations. For example, Ni et al. [1994], with support from the Office of Naval research, have shown that methanol and ethanol can reduce soot concentrations in ethylene flames, above and beyond the simple effect of diluting the fuel. The mechanisms responsible for this effect have not been identified, and will be the subject of our experiments in alcohol-doped flames.

We have also begun experiments with air-diluted flames to test the impact of the degree of premixing on hydrocarbon growth. Partial premixing can occur in jet engines due to turbulent mixing and the use of air in the fuel atomization process. Our results show that small degrees of premixing can significantly affect the sooting tendency of unsaturated fuels because molecular oxygen and O-atoms can react with the parent fuel and its immediate decomposition products via molecular rearrangement reactions that greatly expand the product distribution [McEnally and Pfefferle, 1999b].

3 Description of Experimental Techniques

3.1 Fragmentation-Fluorescence Measurements of Acetylene

In this technique, the intense 193 nm ArF output of an excimer laser is used to photodissociate acetylene into excited state CH and C_2 molecules, and then detect the fluorescence that occurs as these fragments relax to their ground states. Raiche et al [1989] have successfully measured an acetylene profile in a flame using a dye laser tuned to 215.9 nm as the excitation source, and C_2 fluorescence from 400 to 600 nm for detection. By using the excimer laser output at 193 nm as an excitation source, we have far more laser energy to work with and avoid the difficulty of operating a dye laser at very short wavelengths.

The 193 nm ArF excimer laser output is ideal because it is the shortest-wavelength (highest-energy) laser line that can propagate through air. The photon energy at 193 nm is 620 kJ/mole, which is substantially higher than hydrocarbon bond strengths (350 to 450 kJ/mole); thus a single 193 nm photon is energetic enough to produce photodissociation. Furthermore, because the output is very intense, mild focusing produces photon densities that are high enough to drive multiphoton absorption, such that extensive fragmentation will occur in the target gas.

Room-temperature studies have shown that exposure of acetylene to 193 nm light produces banded fluorescence throughout the 190 to 1100 nm spectral region from CH and C_2 fragments in excited states [Jackson et al., 1978; McDonald et al., 1978]. Among the specific bands observed are CH: $A \rightarrow X$ at 431 nm, C_2: $A \rightarrow X$ from 690 to 1100, C_2: $D \rightarrow A$ at 467, 518, 550, and C_2: $C \rightarrow A$ at 340, 369, 385, and 405. Analysis of the laser intensity-dependence and temporal behavior of these bands show that the C_2 and CH are direct photoproducts of the acetylene, and thus their concentration is directly proportional to that of acetylene. This range of fragments and spectral lines creates many detection options for avoiding interferences. The CH and C_2 bands were between 300 and 700 nm and in most cases were not subject to interferences from the oxygen bands.

Our detection equipment includes a 0.3 m monochromator (CVI CM110), photomultiplier tubes, a high speed (100 M samples/sec) digital oscilloscope, and custom software for transferring and processing data from the oscilloscope.

Since the 193 nm output form the excimer laser is so intense and energetic, it can produce many emissions that potentially interfere with the acetylene measurements. Thus an important part of this work is the characterization of these interferences and the development of strategies to avoid them. Guidance from the soot model and our extractive sampling acetylene measurements will greatly assist us in this process. We will also use our ability to independently measure most of the potential interfering species such as other hydrocarbons and soot particles. Furthermore, we have a Nd:YAG-pumped dye laser system available in our laboratory (Spectra-Physics DCR-3G/PDL-2), which can be used to directly measure CH and C_2, to determine whether their background levels in the flame lead to interferences.

3.2 Future Work: Generation of Tunable VUV Light

The second important application of the new excimer laser in our research will be as a source of tunable VUV light.

Currently, our principal technique for analyzing the gases sampled from flames is single-photon photoionization mass spectrometry with a 118.2 nm VUV laser beam [McEnally et al., 1999]. The 118.2 nm light is generated by frequency-tripling the 355 nm output of a Nd:YAG laser in a xenon cell and is not tunable. This technique offers simultaneous, highly sensitive (part-per-million) to nearly all C3 and C12 hydrocarbons with short measurement times (less than a minute). This range of species includes linear pyrolysis products, one-ring aromatics, and two-ring aromatics, making the technique ideal for studying the hydrocarbon growth chemistry relevant to soot formation. Since the response time is short, the technique can also be used to map two-dimensional distributions, such as the benzene results shown in Figure 4, which are needed for testing multi-dimensional flame calculations.

However, the technique is unable to distinguish between different structural isomers. For example, the concentrations in Figure 4 are really the total of all C_6H_6 isomers, including fulvene and linear compounds as well as benzene. Although benzene is undoubtedly the dominant isomer, for studies of hydrocarbon growth pathways isomer selectivity in many situations such as this would be useful. Fulvene is an intermediate in some proposed benzene formation pathways, such as propargyl self-reaction [Marinov et al., 1996], but not in others, such as acetylene addition to vinylic-radicals [Wang and Frenklach, 1997]; thus measurements of fulvene concentrations would help to determine the relative importance of these pathways.

An ideal approach for obtaining isomer-selectivity in photoionization mass spectrometers is to use a tunable VUV laser beam as the photoionization source and to tune it in-between the ionization energies of the isomers in question. The spacing between the ionization energies of typical structural isomers is wide enough for them to be easily discriminated; for example, the ionization energies of benzene and fulvene are 9.25 eV (134.2 nm) and 8.36 eV (148.6 nm). A great advantage of this approach is that the only species property needed to apply it is the ionization energy, which is known for virtually all relevant hydrocarbons [Lias et al., 1988].

Most methods for generating tunable VUV light are difficult and tedious to use however, Strauss and Funk have demonstrated a technique based on four wave difference frequency mixing of 193 nm excimer laser radiation with dye laser radiation in a cell containing H_2 or Kr [Strauss and Funk, 1991]. They generated light that was continuously tunable from 117 to 150 nm (10.6 to 8.3 eV), a range which includes the ionization energies of most hydrocarbons relevant to soot formation, and reported that the upper-wavelength limit could likely be extended to nearly 193 nm given additional spectral coverage to the dye laser. With the addition of an excimer laser, this method of VUV generation could be implemented in our laboratory without additional equipment, since we already have a dye laser system in place.

We could then immediately make isomer-selective photoionization measurements since the rest of the mass spectrometer (vacuum system, molecular beam generator, mass filter, ion detector) does not have to change.

4 Summary

The requested excimer laser is in use and is performing well. Initial studies of acetylene measurement show minimal interferences in the lightly sooting methane flame. The CH signal spatially follows acetylene measured by our in-situ MS sampling technique. This is important because it confirmed inadequacies in the chemical mechanism used in our soot model developed by Smooke/Hall/Colket in conjunction with our group. New mechanism analysis has produced significantly improved predictions. This work will be submitted to the upcoming International Symposium on Combustion.

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Part 2: Financial Report

Item(s)	Vendor	PO #	Amount
Compex 205 Excimer Laser Multigas with Halogen Fluorine Source	Lambda Physik	YKP1037684	$116,900.00

Cost Sharing Component from Yale Accounts:

Item(s)	Vendor	PO #	Amount
PMT Detector and Acc.	Oriel Instruments	1039072	$1,042.00
UV Excimer Optics	CVI Laser Corp	1038555	$920.00
Energy Meter and Acc.	Coherent Inc. – Auburn Group	1038724	$1452.00
High Purity Instrument Regulators	Connecticut Airgas	UNP1000073 (R61630)	$569.00 $569.00
Laser Safety equipment	Newport Corporation	1038637	$340.00
Optics Table and Table Legs	Newport Corporation	1038155	$3.529.80
UV Optics	Thurlabs	QO501624	$288.00
Laser Gases	Spectra Gases	1038151	$1,361.00
Preamplifier	Stanford Research Systems, Inc.	1038641	$1,100.00
Dell Dimension XPS 700 MHz Pentium III	Dell Computer	1042322	$1,932.00

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oller		YNP1048599
LeCroy Model LT342 $7,474.00 2 Channel Waverunner Color Oscilloscope and software	LeCroy Corp	1030647