## Paper # 715353 Measurement of Diatomic Oxygen in the Exhaust Plume of a Mini-Hybrid Rocket Philip A. Ashley

#### Abstract

Hybrid rockets have advantages over liquid or solid rockets because they are safer and can be stopped and restarted. The goal of these studies are to characterize combustion efficiency and environmental impact of hybrid rocket motors. The design and testing of diode laser sensors to measure chemical constituents in exhaust plumes of hybrid rockets is presented. A diode laser with output in the 763 nm region of the oxygen spectrum was employed to measure oxygen concentrations in exhaust plumes of mini-hybrid rockets. The temperatures of the exhaust plumes were estimated to be 3000 K. This high temperature reduces the sensitivity of oxygen measurements because the oxygen molecules are spread over a much greater number of energy states than when at 300 K. Thus, there are fewer molecules to absorb radiation at the measuring wavelengths. A lock-in amplifier, using second harmonic amplification, was used to measure the signal. Qualitative results showed a 50 mV signal, buried in 75 mV noise, when amplified, produced a 1.0 volt signal with 150 mV noise.

#### Introduction

Diode laser spectrometers are powerful instruments for probing the chemical and physical properties of the exhaust plumes of hybrid rocket engines. This study has two goals. The first is to measure oxygen, hydroxyl radical, water vapor and carbon dioxide concentrations produced by these rocket engines in an effort to enhance their combustion efficiency and thrust capability. The second is to measure the effect hybrid rockets would have on the environment. Information that can be learned from these measurements includes pressure, temperature, combustion efficiency, molecular velocities and number densities of important species. The diode lasers we plan to employ emit in the near infrared region of the electromagnetic spectrum. This region of the spectrum yields signals much less intense than those in the infrared region. However, diode lasers that currently emit in the infrared require cooling to liquid nitrogen temperatures for operation making them less attractive for sensors that are rugged and portable in the field. The negative aspect of using lasers that emit in the near infrared is that the radiation consists of overtone and combination bands from the infrared and these bands are very temperature sensitive. Because of this, diode lasers tuned to wavelengths that measure water vapor, oxygen, hydroxyl radical and carbon dioxide at room temperatures may be useless at the estimated 3000-4700 K produced by hybrid rocket motors. We are currently modeling the spectra of water vapor, oxygen, hydroxyl radical and carbon dioxide in order to define measurement wavelengths that can be employed with a reasonable chance of success. This study examines the use of diode lasers to measure oxygen in the rocket plume.

When the temperature of a gaseous sample, such as oxygen, is raised, the spectrum broadens and the intensities of individual spectral lines are reduced. This is explained by the Boltzmann distribution of energies, defined in Equation 1.

$$n_{i} = g_{i} \frac{n_{0}}{g_{0}} e^{-\frac{\epsilon_{i} - \epsilon_{0}}{kT}}$$

Although the area under the curve of all the spectral transitions is a constant, at high temperatures many more energy levels are occupied and consequently any single transition has a smaller contribution to the overall spectrum. Because of this great diminishment of signal, lock-in amplifiers and harmonic detection schemes must be employed to rescue the signal from the noisy environment of the exhaust plume.

A very positive feature of this research is that, not only do these measurements apply to the exhaust plumes of hybrid rocket motors, the measurement scheme can be applied to any combustion process: truck and automobile engines, airplane engines, power plant chimneys and incinerator plumes. Diode lasers are non-invasive, occupy a small footprint and require a minimal power supply. By including these sensors in combustion processes, feedback loops can be designed to control the combustion process to optimize performance and minimize pollution of the environment.

#### Experimental

Two single-mode VCSEL diode lasers, obtained from Laser Components GmbH were employed in all reported studies. The lasers were used to probe the oxygen spectrum in the region of 759 to 766 nm (See oxygen spectrum in Figure 1).



Figures 1 and 2. Left-hand figure is the HITRAN-PC produced spectrum of diatomic oxygen at 296 K at a path length of 1.00 m. The diode lasers obtained from Laser Components produce emission wavelengths in the range 759 to 766 nm. The right-hand figure shows the spectral features that could be observed in the accessible region of our lasers.



Figures 3 and 4. Oscilloscope traces of oxygen measurements of laboratory air over an open path. The two oxygen peaks centered around 763 nm are visible in both figures. Figure 4 on the right is an optimized spectrum of the one pictured in Figure 3 after expanding the time base to 0.2 milliseconds per division and increasing the path length between laser and detector. Figure 4 shows the applied triangular ramp on Channel 2 of the oscilloscope. Each oscilloscope trace repeats the spectrum as the current ramp sweeps positive and negative across the set point current of the power supply. The repeated spectra are mirror images of each other.

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Important parameters for the VCSEL lasers used are summarized in Table 1. Notice the very small tuning capabilities as a function of either temperature or current. A ten degree temperature variation allows tuning over a wavelength range of 0.6 nm while a 2 mA current sweep would allow the laser to be tuned 0.8 nm. This is why lasers used for spectroscopic measurements must be custom fabricated, or hand picked from a stock of units. Figure 5 is a schematic of the measurement apparatus.

Parameter	Symbol	Conditions	Min	Тур	Max	Units
Threshold Current	I <sub>th</sub>		1.0	2.0	4.0	mA
Operating Current	I <sub>op</sub>	$I_{op} = 1.5 \text{ x } I_{th}$		3		mA
Emission Wavelength	Λ	$I_{op} = 1.5 \text{ x } I_{th}$	759	763	766	nm
Optical Output Power (SM)	P <sub>sm</sub>		0.3	0.5		mW
Beam Divergence	Θ	FWHM		12		
Bandwidth	f <sub>3dB</sub>			100		MHz
Linewidth	ΔV	$P_0 = 0.3 \text{ mW}$		30		MHz
Temperature Tuning Coefficient	δλ/δλ			0.06		nm/K
Current Tuning Coefficient	δλ/δΙ			0.4		nm/mA

**Table 1.** Important parameters for VCSEL diode laser used in these studies.



Figure 5. Schematic of apparatus used to measure oxygen. A stainless steel cell was constructed with dimensions 38 mm diameter by 1150 mm long and having 25.4 mm diameter sapphire windows to contain the oxygen and adjust its pressure as needed for these studies. The temperature of the laser was controlled by a Thorlabs TEC 2000 Temperature Controller attached to a Thorlabs Model TCLD-M9 TEC LD Mount which housed the diode laser. For some of the studies, the laser temperature was modulated by imposing a signal from one of the signal generators onto the set point temperature voltage of the temperature controller. The current to the laser was provided with a Thorlabs LDC 500 Laser Diode Controller. The laser current in some experiments was modulated by imposing a sawtooth waveform,  $\sim 20$  Hz, onto the set point current of the laser controller. This was accomplished with an INSTEK Model GHG-8020H Function Generator. When the lock-in amplifier was used, a small, high frequency sine wave, ~20 kHz, was added to the laser current. The sine wave was produced by a Stanford Research Systems Model DS335, 3.1 MHz Synthesized Function Generator. These modulated diode laser signals were detected by a Thorlabs PDA55 Silicon Photodiode Detector by direct measurement with a LeCroy 9310 Dual 300 MHz Digital Storage Oscilloscope. For some of the measurements, the silicon photodiode signal was fed into a Stanford Research Systems Model SR830 DSP Lock-In Amplifier and the amplified signal, or one of its harmonics was measured.



Figure 6.

Figure 7.

Figures 6 through 8 shows different ways of employing a lock-in amplifier when making spectral measurements using diode lasers. Each figure gives the results of measuring ambient laboratory oxygen concentrations over an open path of 2.225 m at 296 K. The bottom oscilloscope trace in each figure is the signal obtained by connecting the silicon diode photo detector directly into Channel 2 of the oscilloscope. Figure 6 shows the advantage gained by passing the signal from the photodiode detector through the lock-in amplifier and measuring the first harmonic of the photo detector signal. The lock-in amplified signal is connected to Channel 1 of the oscilloscope. The sweep rate of the oscilloscope is such that the same oxygen peak appears three times on the oscilloscope screen. The oxygen produces a peak of approximately 50 mV amplitude when observing the unamplified signal on Channel 2. If we calculate the noise in the signal as one-fifth of the amplitude of the trace being equal to one standard deviation, the noise is calculated to be 3 mV. This yields a signal to noise ratio of 16.7. The amplitude of the first harmonic of the oxygen peak measured after being processed by the lock-in amplifier is approximately 10.05 volts and exhibits a one standard deviation noise level of around 0.2V. The signal to noise ratio of the amplified first harmonic is 50.3 almost exactly three times better than the unamplified signal and a signal

201 times larger. Figure 7 shows oxygen measurement under the same conditions as the measurement discussed in Figure 6. This time the lock-in amplifier is used to amplify the second harmonic of the signal received from the photodiode detector. Almost exactly the same noise levels and signal levels were achieved as before. However, there is no simple relationship between the signal trace of a second harmonic signal and the concentration of oxygen and significant mathematical manipulations must be carried out to obtain a value proportional to concentration. See J. Reid and D. Labrie, Second-Harmonic Detection with Tunable Diode Lasers - Comparison of Experiment and Theory, Applied Physics, 1981, B 26, 203-210, for an excellent discussion of the topic. Figure 8 is a measurement of laboratory oxygen under the same experimental conditions as Figures 6 and 7. This time the third harmonic of the amplified silicon photo detector is passed to Channel 1 of the oscilloscope. Our assessment of these three methods indicates no real advantage for going to additional trouble of using second or higher harmonics when measuring oxygen when the amount of oxygen is substantial. These methods do yield a significant advantage when measuring much smaller concentrations, however.

Figure 8.

Graph 1 shows the effect on the oxygen signal when the pressure of oxygen is varied from 780.7 to 27.8 torr. These measurements were carried out in the apparatus illustrated in Figure 5. As the pressure is lowered the intensity of the signal becomes smaller and narrower. The decrease in peak width is due to a reduction of pressure broadening of spectral lines. Doppler broadening, due to the range of velocities exhibited by the gas molecules at right angles to the measurement axis, is still present.



Figure 8. Oscilloscope traces of oxygen measurement before rocket is fired.

The great advantage of using a lock-in amplifier to measure oxygen concentrations becomes very apparent when trying to measure through the noisy environment of the rocket exhaust plume. Figure 8 shows the oxygen concentration, as measured directly from the silicon photodiode detector being connected to the oscilloscope. This is recorded in the oscilloscope as Trace C. The oxygen detected is the oxygen in the laboratory between the diode laser source and the diode laser detector with the rocket motor off. The distance between laser and detector is 2.225 m. Notice the superposition of the 20 kHz modulation frequency on Trace C. This 20 kHz frequency is the one "locked on" by the lock-in amplifier and only noise and signal at this frequency is amplified. The amplitude of the oxygen signal is 0.45 volts for unamplified Trace C. Trace D, which is the amplified first harmonic signal from the lock-in amplifier, is shown at the top of Figure 8. The amplitude of the oxygen trace from the lock-in amplifier corresponds to 6.0 volts, a gain of 13.3 over the unamplified signal. Figure 9 shows what happens when the rocket motor is being fired and the oxygen is measured. The oxygen measured includes the laboratory air between the detector and laser as well as the oxygen in the rocket plume. The unamplified signal from the photo detector is recorded on Channel 1. As can be seen, it is virtually impossible to pick an oxygen signal from the noisy environment of the rocket motor exhaust plume. The first harmonic of the lock-in amplified photo detector signal is recorded at Channel 2 of the oscilloscope. The oxygen signal is easily measured.

Although we have successfully measured, in a qualitative sense, the oxygen in the plume of a lab scale mini-hybrid rocket, much challenging work lies ahead. Now that we have the basic equipment set up and operating, we plan to work diligently on reducing noise in the entire measurement system. We plan to implement National Instruments LabVIEW software and hardware to allow us greater freedom in designing the experiments needed and recording the data. Much work needs to be done in taking the raw data and calculating a spectral line shape with sufficient accuracy to be able to do some curve fitting required to estimated pressure and Doppler broadening, temperature and number density of the oxygen in the plume. This is an exciting, challenging project and we are looking forward to working our way to a successful implementation of using diode lasers as sensors for hybrid rocket exhaust plumes and a host of other combustion plumes.

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Wavelength, Arbitrary Units

Graph 1. Oxygen signal versus oxygen pressure

Figure 9. Oscilloscope traces of oxygen measurement during rocket firing.

### Conclusion