DEMONSTRATION OF A RAPIDLY STRAIN TUNED Er\textsuperscript{3+} DOPED FIBER LASER FOR SENSITIVE GAS DETECTION

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*Applied Optics* 38(15) 3370-3375 (1999)

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DEMONSTRATION OF A RAPIDLY STRAIN TUNED Er$^{3+}$ DOPED FIBER LASER FOR SENSITIVE GAS DETECTION

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Abstract

This paper describes a novel combination of a diode-pumped, wavelength modulated Er$^{3+}$ doped fiber laser light source with a sensitive noise cancellation circuit for detection of acetylene and ammonia. The laser tuning element, a fiber Bragg grating, was mounted in such a way that it could be strained controllably and rapidly, allowing noise cancellation techniques to be applied to the wavelength modulated output of the fiber laser. The experimental setup is relatively simple and can be extended to other fiber laser wavelengths where semiconductor lasers are not readily available by selecting a different fiber Bragg grating and gain medium.

OCIS Codes: 140.3510, 140.3600, 300.1030.

1. Introduction

Highly sensitive techniques for the measurement of gas concentrations by absorption spectroscopy have been a subject of interest for many years. In previous publications, we have demonstrated highly sensitive absorption measurements using a balanced detection scheme which
allows near shot-noise-limited measurements without high-speed wavelength modulation techniques.\textsuperscript{1,2} In this work, we adapt this technique to sensitive absorption measurements using fiber lasers where the laser wavelength is tuned repeatedly and controllably at bandwidths up to a few hundred Hertz.

Fiber lasers are attractive for absorption spectroscopy because they can be operated at wavelengths where semiconductor lasers are not readily available. For example, rare earth doped heavy metal fluoride glasses have been demonstrated in the 2 to 3 $\mu$m region. This wavelength region contains strong overtone transitions from technically important species such as CO and NO. Specifically, Tm$^{3+}$ lasers have been demonstrated from 1700 to 2015 nm in silica or fluoride fibers and from 2250 to 2400 nm in fluoride fibers.\textsuperscript{3} Lasers based on Ho$^{3+}$-doped fluoride fibers have been demonstrated from 2 to 4 $\mu$m.\textsuperscript{3,5}

Although near-IR fiber lasers have been available for several years, they have only shown limited use in absorption spectroscopy due to a lack of controlled tuning methods. All-fiber tuning methods based on temperature tuned fused couplers have been demonstrated with tuning ranges over 40 nm, although the slow response of this method make it unsuitable for rapid scanning.\textsuperscript{6,7} Sputter deposited piezoelectric coatings on a fiber Bragg grating have also been investigated.\textsuperscript{8} The feasibility of temperature tuning of a fiber Bragg grating in a Tm$^{3+}$ doped silica fiber laser cavity has been demonstrated, where the laser was used to detect methane near 1.68 $\mu$m.\textsuperscript{9} More recently, an external grating tuning mechanism has been used in a Tm$^{3+}$ doped fluoride fiber laser to detect methane at 2.31 $\mu$m.\textsuperscript{10}

We demonstrate here an all-fiber wavelength modulation strategy for tuning at rates up to several hundred Hertz. This is combined with a sensitive balanced detection technique to achieve
a detection sensitivity comparable to a conventional diode laser-based sensor system as a first step in the eventual development of longer wavelength sources using the materials described above. The technique is demonstrated here in the near-IR using an Er$^{3+}$ doped fiber laser. To obtain quantitative results and to gather more information about the fiber laser’s properties, we compared our results with those we obtained with an external cavity diode laser.

Various properties of the tuning method and fiber laser were investigated with regard to applications to gas sensing. Among them are static and dynamic tuning rates, the effect of multi-longitudinal mode operation, and the ultimate degree of noise cancellation possible using a balanced detector. We focused our spectroscopic studies on acetylene and ammonia as they have absorption lines accessible to an Er$^{3+}$ doped fiber laser operating near 1.55 µm.

2. Experimental Setup

The experimental setup used in this investigation is shown in Figure 1. The fiber laser was pumped by a 975 nm pigtailed, grating stabilized laser diode. The maximum pump output power was approximately 100 mW. The fiber coupled pump radiation was then fusion spliced onto a 2 x 2 fused coupler designed to have a 3 dB split ratio at 1550 nm. About 75% of the pump radiation was coupled into the fiber laser while the remaining power was embedded in index-matching gel. The fiber laser cavity itself consisted of a fiber Bragg grating (FBG), a 20 cm length of fiber with an Er$^{3+}$ doping level of 3500 parts in $10^6$ and a 65 cm long gold tipped section of single mode telecommunications fiber. There were large core to core mismatches between the fiber containing the Bragg grating (7 µm core diameter) and the active fiber (4 µm core diameter) resulting in a large cavity loss. The typical output power of the fiber laser was on the order of 10 µW. Ideally, the output power of the fiber laser cavity could be increased to the milliwatt range if the core
mismatch losses between the active fiber and the other two fibers (grating fiber and mirror fiber) were eliminated and the fiber were designed to maximize coupling from the pigtailed pump laser diode. Such an increase in power would improve the shot noise limited detection; but as we will show here, the sensitivity is usually limited first by weak interference fringes from elements in the system which cannot be cancelled by balanced detection. When using a low noise current source to excite the pump diode at 980 nm, we have observed fluctuations of ~4% in the output power of the fiber laser when detected over a 100 ms period and this noise level was independent of the tuning rate of the laser. At the present time, the source of this amplitude noise is not well understood and may be related to mode competition or opto-mechanical instabilities in the laser cavity.

The FBG was written with a 1.07 μm period phase mask which, for the fiber used (n_{eff} = 1.449 at 1550 nm), corresponds to a grating reflective wavelength of 1551 nm. This was a phase mask that we had on hand and was not the ideal period phase mask for detection of acetylene or ammonia. The effective refractive index, n_{eff}, of the fiber was found from the Bragg condition for peak reflection given below where Λ, the index modulation period in the fiber, is one half the period of the phase mask:

\[ \lambda_B = 2n_{eff}\Lambda \]  

(1)

If the fiber is exposed to the excimer UV laser pulses while in tension, the period of the index modulation and the peak reflective wavelength will be reduced when the tension is removed.\textsuperscript{11} We use this effect to produce a grating at a slightly shorter wavelength because the acetylene absorption lines at room temperature are most intense in the 1530 to 1540 nm range.
The grating used in the experiment had a peak reflectivity of 90% near 1547.5 nm and a Full Width Half Maximum (FWHM) of 0.3 nm. This reflectivity was measured actively during exposure to the UV pulses by launching a 1550 nm LED into the fiber being exposed and measuring the transmitted light with an optical spectrum analyzer.

The spacing of the resonant longitudinal modes, which is a function of the cavity length, is approximately 120 MHz. Since this spacing is less than the FWHM of the FBG reflectivity spectrum, the fiber laser oscillated on more than one longitudinal mode. Thus, the absorption lineshape obtained by scanning the fiber laser across an absorption line will be source broadened in addition to the Doppler and collisional broadening. As described below, this laser linewidth was extracted from the measured absorption lineshapes assuming a nominal Gaussian laser profile.

The output of the fiber laser was taken from the remaining port of the 2 x 2 coupler. It was passed through an isolator, collimated and split into signal and reference beams which were detected by InGaAs photodiodes. The photodiodes were incorporated into low-noise Balanced Ratiometric Detector (BRD) system described in detail in previous publications.\textsuperscript{1,2,12} A 50 cm long absorption cell was filled with varying gas pressures of the species of interest and absorption scans were recorded.

3. \textbf{Wavelength Modulation Technique}

In order to sweep the laser wavelength across the absorption line shape, we relied on the fact that the FBG center wavelength is sensitive to temperature and strain.\textsuperscript{13} As illustrated in Figure 2, one end of the grating was immobilized in epoxy while the other end was attached using the same epoxy to a preloaded PZT actuator (a multilayer PZT stack capable of extension at low applied voltages) with a total extension range of 90 µm. The epoxy (Tracon BAF120) we used
was the same epoxy that is commonly used to fix fiber in ceramic ferrule connectors. The epoxy was allowed to cure for 12 hours before tension was applied. This method of straining the grating allowed the fiber laser to be tuned across approximately 3 nm or 15 cm\(^{-1}\). It was possible to dither the laser about a certain wavelength at a rate of several hundred Hz by applying a triangle wave to the PZT driver. The static (dc) tuning curve is shown in Figure 3 and was obtained using an optical spectrum analyzer. From this data, a static tuning coefficient of approximately 4 GHz/ V is deduced.

To investigate the effect of the modulation on the tuning rate, the fiber laser's output was observed at various modulation frequencies with a scanning Fabry-Perot interferometer. The resolution of the interferometer was not sufficient to resolve the individual longitudinal modes, but it was possible to monitor the total wavelength excursion of the unresolved group of oscillating modes as a function of frequency. We found that triangle waves of 1, 2, 5 and 10 Hz all gave tuning coefficients of approximately 2.6 ± 0.2 GHz/V. This reduction in the dynamic tuning rate compared to the static value was also verified by matching the observed acetylene absorption features to their known positions.\(^{14}\) This reduction cannot be attributed to the PZT actuator itself; we believe it arises from the compliance of the epoxy because we have also observed that the overall tuning range increases with the stiffness of the glue.

4. Absorption Measurements

The absorption cell was filled with pure acetylene to a pressure of 56.6 Torr. The PZT drive voltage was scanned with a 10 Hz, 4 V p-p triangle wave which was added to a dc drive voltage of about 40 V. Figure 4 (a) shows the raw digital oscilloscope data obtained from the BRD as a function of the ac voltage applied to the PZT. The absorption features appear as dips in
the BRD output. The data points are connected with solid lines for the purpose of displaying a large number of points (528) without cluttering the plot. Figure 4 (b) shows reduced absorption lineshape data; the solid curve is obtained with the wavelength modulated fiber laser (raw data shown in Figure 4 (a)) and the dashed curve was obtained with a wavelength modulated single frequency External Cavity Diode Laser (ECDL; New Focus Model 6262) at a reduced acetylene pressure of 16.9 Torr. Both curves have been baseline corrected and the absorption axis of the ECDL trace has been scaled for easy comparison with the fiber laser trace. The linewidth of the ECDL is less than 1 MHz and shows the Doppler-limited absorption lineshape whereas the lineshape acquired with the fiber laser includes both laser and collisional broadening contributions.

The stronger absorption line in the fiber laser scan has an absorption of 10%. We have assigned the stronger of the two observed absorption lines to be the P(35) line of the $v_1 + v_3$ combination band of $^{12}$C$_2$H$_2$. This band is denoted by 1010$^00^0$--0000$^00^0$ and we will call it Band A. The second, weaker line is the P(28) line of the $^{12}$C$_2$H$_2$ hot band denoted by 1010$^1e$-- 0000$^1e$ which we call Band B. The P(35) line is centered at 6459.0938 cm$^ {-1}$ and is predicted to have a Voigt (FWHM) linewidth of 0.026 cm$^ {-1}$ at 56 Torr, using the pressure broadening coefficient given in the HITRAN database for the fundamental $v_3$ absorption lines.$^{15}$ Note that the ECDL trace shows one additional very weak line on the short wavelength side of the P(35) line. This line is not resolvable by the fiber laser but the slight asymmetry in the fiber laser scan of the P(35) line can be attributed to this unresolved feature.
Using the spectral atlas and line listing in References 14, 16 and 17, we were able to apply a frequency scale to the absorption traces obtained as a function of PZT drive voltage. This was accomplished by matching the known 4.48 GHz frequency difference between the P(28) line of Band B and the P(35) line of Band A to our measurement.

To compare our observed absorption measurements to the fundamental transition linestrengths and thereby verify the quantitativeness of the sensor measurements, we need to have an estimate for the laser lineshape. This can be done by direct measurement with a high resolution Fabry-Perot interferometer or it can be inferred from the absorption lineshapes that are being scanned. We chose the latter method and assumed that the fiber laser oscillates on many longitudinal modes with a Gaussian intensity distribution. Each longitudinal mode was modeled as a Gaussian with FWHM of 5 MHz. This estimate corresponds to the width of longitudinal mode beat signals we have observed with a high speed photodetector and a radio frequency spectrum analyzer. To determine the laser FWHM, we numerically forced a Gaussian intensity distribution of longitudinal modes to minimize the error between the convolution of the laser distribution with the known Voigt profile and observed feature.

Figure 5 compares the best fit convolution profile to the observed feature with a laser linewidth of 650 MHz (0.022 cm⁻¹). The slight deviation from the theoretical profile in the wings is attributed to the unresolved feature shown in Figure 4 which was not included in the analysis. With this laser lineshape, we can convert absorption cross section, \( \sigma \), to linestrength through the relation

\[
S = \frac{\sigma}{g_{\text{obs}}(v_0)}
\]  

(2)
where the units for the absorption cross section are cm$^2$/molecule and the units for $g_{\text{obs}}$ are in centimeters. For the 56.6 Torr measurement, we can estimate the cross section for the 10% absorption at the peak of the P(35) line to be $(1.2 \pm 0.5) \times 10^{-21}$ cm$^2$/molecule, which for the source broadened Voigt lineshape gives a linestrength $S = (5.2 \pm 0.5) \times 10^{-23}$ cm/molecule where we have taken into account the line broadening due to the laser's longitudinal mode spectrum by dividing the absorption cross-section by $g_{\text{obs}}(v_0) = 22.93$ cm. This value was determined from the theoretical expression for the observed lineshape after estimating the average laser lineshape. The same line using the ECDL exhibited an 8% peak absorption at 16.9 Torr which gives a linestrength of $(6.5 \pm 1.0) \times 10^{-23}$ cm/molecule. In this case, $g_{\text{obs}}(v_0) = 46.2$ cm and the single frequency laser is treated as a delta function because its width is much smaller than the width of the feature. The linestrengths obtained from both lasers agree reasonably well and indicate that the absorption recorded from the fiber laser is absolutely quantitative. We note that if extremely high resolution measurements are required, thelinewidth could be narrowed further (at the expense of system and tuning complexity) by introducing an intracavity etalon or using a distributed feedback structure. In this paper, however, we focus on the simplicity and utility of a slightly multimode fiber laser.

When the absorption cell pressure is reduced to 900 mTorr, the fiber laser absorption spectrum appeared as in Figure 6. This is again with respect to the P(35) rovibrational line of Band A. The S/N ratio for this single scan was approximately 74 if we consider the small scale variations and approximately 8 if we consider the larger variations believed to arise from unsuppressed étalons in the system. The residual reflections from the anti-reflection coated windows forming the absorption cell are likely the predominant étalon source. As a conservative
measure, we will choose the smaller S/N ratio of 8 to predict an ultimate absorbance sensitivity for the fiber laser. It follows from the measured peak absorption of $3.25 \times 10^{-3}$ and the 470 Hz detection bandwidth that the minimum detectable absorption will be $4.1 \times 10^{-4}$ or $1.9 \times 10^{-5}$ Hz$^{1/2}$ for a S/N ratio of 1. This value is comparable to a typical limit for a fiber-coupled current-tuned laser based sensor$^{18}$ and to previously reported values using an ECDL.$^{19}$

Given that the fiber laser output power was on the order of 10 µW, and the photodetector had a responsivity of 0.75 A/W, we calculate that the shot-noise-equivalent absorbance is $2.9 \times 10^{-7}$ Hz$^{-1/2}$ and we see that we are a factor of 65 above the shot noise limited absorbance. This excess noise is attributed to the etalons in the optical system rather than to the performance of the laser or detector system and could presumably be lowered using an improved setup.

To evaluate the effect of pressure broadening on the sensor response, we filled the absorption cell to 49 Torr with C$_2$H$_2$ and then added nitrogen until a total pressure of 777 Torr was attained. The measured linewidth at 777 Torr was 2.5 GHz or 0.083 cm$^{-1}$, from which we determine a nitrogen pressure broadening coefficient (FWHM) of 0.066 cm$^{-1}$/atm. In this case, the laser linewidth is less than that of the absorption feature so it has a small effect in determining the linewidth at 1 atm. For a fixed small number of absorbers (i.e., the regime where self-broadening is negligible), we can compute $g_{\text{obs}}(v_0) = 8.8$ cm for a 1 atm nitrogen broadened line. In comparison, the same line observed in a low pressure Doppler broadened regime will only show Doppler and source broadening and $g_{\text{obs}}(v_0) = 37$ cm. Because the peak value of absorption equals $SNL g_{\text{obs}}(v_0)$, it follows that concentrations near the sensitivity limit of the fiber gas sensor will have peak absorptions reduced by a factor of 4.2 over their Doppler values. If we use an average linestrength from our measurements, we can compute an ambient pressure detection limit.
of 15 ppmv-m (parts in 10⁶ by volume in a 1 m path) operating on this line with a 1 Hz bandwidth.

The particular absorption line accessed with the present laser is approximately 200 times weaker than stronger lines originating from rotational levels with peak population at room temperature. For example, the P(11) line of Band A at 1531.59 nm would be easily accessible with another fiber laser and would allow a detection limit of 75 ppbv-m (parts in 10⁹ by volume in a 1 m path) using the present setup.

Finally, we applied the laser to detect NH₃ which also has many absorption lines in the operating region of our laser.²⁰,²¹ The observed lineshape for a line near 1548 nm after baseline correction is shown in Figure 7 and the peak absorption is 5.8%. For this measurement, the pressure in the 50 cm long cell was 19.7 Torr and the data was acquired at room temperature. The integrated absorption signal is shown in Figure 8 as a function of NH₃ concentration in the cell. We attribute the deviations at low concentrations to unreliable pressure measurements in the presence of unsteady adsorption/desorption effects in the cell. If we assume a pressure self-broadening coefficient of 0.8 cm⁻¹/atm, a common value listed by HITRAN for the fundamental band,ν₃, we can deduce a linestrength of (9.5 ± 1) x 10⁻²³ cm/molecule. This value is found from the measured peak cross section at 19.7 Torr and takes into account laser linewidth. For comparison, we also obtained a linestrength for this line using the single frequency ECDL. With the ECDL, the peak absorption is about 2% for a pressure of 2.6 Torr and g_{obs}(ν₀) = 43.07 cm. This gives a linestrength of (11 ± 1) x 10⁻²³ cm/molecule which shows reasonable agreement with the fiber laser. The line that we measured has been observed at 6459.459 cm⁻¹, but has not been spectroscopically assigned so there is no theoretical value with which to compare our
measurements. We also note that this was the strongest line that we could find in the three nanometer tuning range of the fiber laser. Using the value of minimum detectable absorption obtained from our acetylene scans, we predict a detection limit at ambient pressure of 18 ppmv-m on this line.

5. **Ultimate Sensitivity And Conclusions**

For the laser system we used to demonstrate the technique, any molecule which absorbs in the Er$^{3+}$ gain region may be detected simply by selection of an appropriate fiber grating. Sensitivities in ambient air on the order of 10's ppmv-m are predicted for several gases which absorb under the Er$^{3+}$ bandwidth. The strongest lines with linestrengths of $2 \times 10^{-21}$ cm/molecule or greater should give detectivities of below 1 ppmv-m. This technique can be combined with multipass cells if higher detectivity is required. Quantitative spectroscopic information was obtained using a reasonable estimate for the lineshape of the fiber laser. In addition to acetylene and ammonia detected here, there are other gases which have absorption lines in the region accessible to an Er$^{3+}$ fiber laser; they include CO$_2$, OH, CO, HCN and HI. Numerous additional species such as NO and N$_2$O and H$_2$CO are accessible with Tm$^{3+}$ fiber lasers in silica glass.

We believe this combination balanced detector and wavelength modulated fiber laser may have general appeal for compact sensitive gas sensors when applied to wavelengths where conventional diodes are not available. Species having strong lines in wavelength regions accessible to fluoride fibers should also benefit from this all-fiber rapid wavelength modulated balanced detector configuration. In a well designed fluoride fiber laser system, diode pumping should be possible, allowing the potential for compact gas sensing in wavelength regions inaccessible to diode lasers.
Acknowledgment

The authors would like to acknowledge Lucent Technologies Inc. for providing the rare earth doped fiber.
References


Figure 1. Experimental setup used to detect acetylene and ammonia.
Figure 2. Mounting details for PZT controllable Bragg wavelength modulation.
Figure 3. Tuning characteristics of fiber laser under a steady PZT voltage.
Figure 4.  (a) Raw acetylene absorption data obtained from the fiber laser/balanced ratiometric detector with triangular wave PZT wavelength modulation voltage depicted.

(b) Acetylene spectra of P(35) line from Band A and the P(28) line from Band B. Solid curve - fiber laser. Dashed curve- ECDL. Other conditions: T = 296 K, pathlength = 50 cm, and acquisition time = 100 ms.
Figure 5. Theoretical lineshape (FWHM = 650 MHz) which best fits the experimental lineshape of the acetylene P(35) line in Band A. Pathlength is 50 cm.
Figure 6. Observed acetylene absorption spectrum at 900 mTorr in a 50 cm path with S/N estimates. $T = 296$ K and acquisition time = 100 ms.
Figure 7. Ammonia spectrum at 19.7 Torr near 1548 nm in a 50 cm path, with T = 296 K and 100 ms acquisition time.
Figure 8. Integrated absorption signal as a function of ammonia concentration.