Development of sensors using evanescent wave interactions in sapphire optical fibers

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Abstract

The development of tunable diode laser absorption sensors for measurements in industrial boilers, both through direct absorption and evanescent wave absorption have been performed in the work presented here. These sensors use both direct and indirect absorption through the use of evanescent interactions within a coal firing combustion environment. For the direct absorption sensor, wavelength modulation absorption spectroscopy with second-harmonic detection was implemented within a physical probe designed to be placed with the flue stack of a power plant. Measurements were taken of carbon dioxide and water vapor concentration during operation at a local industrial facility. The design of this sensor probe overcomes problems of beam steering and permits a reference gas measurement. Extracted concentration data and design elements from the direct absorption measurements are presented. In addition, development of a sapphire fiber-based sensor using evanescent wave absorption along the outside of the fiber is presented. Evanescent absorption allows for the laser transmission to be maintained in the fiber at all times and may alleviate problems of background emission, beam steering, and especially scattering of the laser beam from solid particles experienced through free path direct absorption measurements in particulated flows. Laboratory measurements using evanescent fiber detection are presented.

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Executive Summary

Process control in coal power plants relies on the availability of sensors for temperature and gas-species concentrations that can survive the high temperature, high pressure, and particulated flows present throughout the plant. Most existing sensors utilize extractive gas sampling which is slow and may be inaccurate for trace pollutants. Some optical sensors are available that have improved time response but these are generally limited to low temperature, or low particle loading regions of the system.

This project examined the feasibility of optical sensors that utilize exposed fiber probes placed directly in the harsh flowfield, consisting of high particulate levels, high temperatures and high, non-uniform velocities. The proposed fiber sensors take advantage of two unique advancements over existing sensors: (1) the use of evanescent-wave optical interactions through the wall of the fiber probe and (2) the use of sapphire fibers. In all fiber transmission, a portion of the electromagnetic field exists outside the fiber core in a so-called evanescent wave. By using a bare sapphire fiber as the sensor medium, a variety of proven laser diagnostic techniques can be used without launching a free laser beam through the combustion environment while also avoiding the temperature limitations created by existing fiber coatings. This alleviates the three primary difficulties with application of laser diagnostics in coal combustors, in particular, flame emission background signal is not collected at the detector, light scattering from solid particulates does not interrupt the laser beam and temperature limitations are significantly extended over current coated fibers. Evanescent-wave interactions have not been used for sensing in combustors due to the low melting temperature of normal SiO_2 fibers. Sapphire fibers give this technique a very high probability of success since sapphire has a high melting temperature and superior resistance to chemical deterioration as compared to SiO_2 and can be used without modification. The evanescent-wave probe will be functionally similar to a thermocouple and can be used through any single access port in a coal power plant, but would provide significant improvements for measurements of temperature and gas composition, including trace pollutants.

During this 1-year project, bare fibers were tested in laboratory gas cells and laboratory scale combustors. The durability of the bare fibers were tested for lifetimes at flame temperatures and the sapphire was able to withstand extended placement into a 1900 K flame. Evanescent absorption was measured through a fiber; however the fiber required tapering, which resulted in a 15 μm diameter probe location. This fiber was found to be very fragile, and for any future implementation of a fiber detector, a structural support or substrate would be required to prevent fiber failure for measurements taking place in high speed flows. Coatings for the bare fiber could also be explored to either enhance durability or add functionality.

During the final period of research a design of a sensor was created which used a direct absorption method for measurement of species concentrations. The sensor developed was tested at a coal power plant run by Alstom Power (Windsor, CT). Actual measurements of CO_2 and H_2O at the plant using different types of sensors helped improve the understanding of requirements and specifications to develop sensors in the future. Sensors tested include various configurations for direct absorption measurements of the gases of interest. Results from these tests are discussed in this report.

Introduction

Active control of air flow rates and staging in coal power plants relies on accurate and fast feedback of operating conditions. Current gas sensors typically rely on physical probes that extract samples after combustion. Laser based sensors have several advantages compared to physical probes including faster response times [1,2] and increased gas selectivity and sensitivity [3-9]. All reported applications of laser diagnostics in coal combustion, e.g. [1], utilize direct optical transmission where a laser beam is launched through the test section using conventional lenses. In coal systems, these direct approaches are severely hindered by optical attenuation caused by solid particulates in the laser beam path, by beam steering due to non-uniform temperature distributions, and by a noise at the detector caused by natural flame emission. Commercial products utilizing direct absorption for coal combustors have recently become available [10], but this approach has not been extended to highly loaded environments such as fluidized bed or high pressure combustors.

The proposed work addresses development of improved sensors that utilize optical fibers placed directly in the flow to be measured. The sensors take advantage of species selectivity and sensitivity available from laser techniques while avoiding low transmission, signal alignment and signal processing problems that plague direct transmission. The enabling technology for this project is newly available sapphire optical fibers, which have high melting temperature (2040 °C) and inherent resistance to chemical attack. Since the sapphire fiber can be placed directly in the hot flow, measurements can be made using light that naturally extends beyond the bare cylindrical surface of the fiber. This mode of interaction avoids problems with launching a free laser beam through the particulate laden flow. No material development or coatings are required; consequently, commercially available fibers can be used ensuring the success of the project and avoiding current temperature limitations for many fiber coatings. The proposed tasks encompass (1) assessment of sapphire fibers for durability in harsh environments, (2) assessment of evanescent wave absorption for gas and temperature sensing in particulated flows, and (3) application of optical sensors in a large-scale coal boiler facility located at Alstom Power (Windsor, CT).

Background

A common technique for gas sensing is tunable diode laser absorption spectroscopy (TDLAS), where the laser beam is collimated and launched through a combustor. As the laser wavelength is tuned over an internal resonance in a species of interest, a portion of the laser emission is absorbed [11]. Since coal-fired boilers have very high radiative emission in the near IR and very low transmission due to scattering from coal dust, the signal-to-noise ratio for direct TDLAS is poor. A few papers have reported direct TDLAS measurements of O2, CO, and H2O [1,2], but each of these measurements have been limited to locations near the heat exchanger in atmospheric pressure devices where the solid loading is reduced. These measurements indicate a clear advantage over exhaust stream physical probes in terms of system response but may not be applicable fluidized bed reactors or gasification systems with larger transmission fluctuations. The proposed work seeks to develop evanescent optical fiber probes for use in harsh environments by avoiding direct laser transmission through the combustor. To enable such sensing, sapphire fibers with unique high-temperature properties will be used to enhance sensor durability.

In optical fiber transmission, the electromagnetic (e-m) field travels through the fiber core. However, up to 40% of the optical power exists outside the core within approximately 1 μm of the fiber surface. This so-called evanescent wave is normally contained within the fiber cladding, but if the fiber is bare or tapered, the e-m field can interact with the gas around the fiber. Evanescent wave absorption spectroscopy can be utilized like TDLAS to measure a broad range of species. It is a well-known phenomenon that has been used for almost 50 years [12-14], but has not been generally applied to reacting flow measurements. The survival of fiber optics in high temperature environments currently uses noble metal coated fibers which fail around 1000-1100° C. In the proposed work, new optical fiber materials that have inherently excellent capabilities to withstand high temperatures will be used to demonstrate evanescent field diagnostics in harsh environments. Commercially available bare fibers are sufficient for these tests.

Approach

During the one year that this project has been funded, experimental equipment for testing evanescent interaction in bare sapphire has been constructed and initial tests have been made. The approach taken is to use tunable diode laser absorption spectroscopy (TDLAS) to measure concentrations of CO_2, as shown in Fig. 1. Inexpensive diode lasers are currently available in the near infrared regions of the electromagnetic spectrum as a result of mass production for the telecommunications industry. In our experiment, the laser is collimated and passed through a gas sample containing carbon dioxide. By tuning the laser to a rovibrational resonance of CO_2, some of the laser radiation is absorbed. Initial efforts were focused on measurements in a calibration test cell. Initial measurements were completed in the first three months of this project and different methods of increasing signal to noise were studied. The way in which the laser was input into the fiber was varied, first through the study of varying the angle of incidence of the input laser beam, and also by changing the surface finish at the entrance of the fiber.

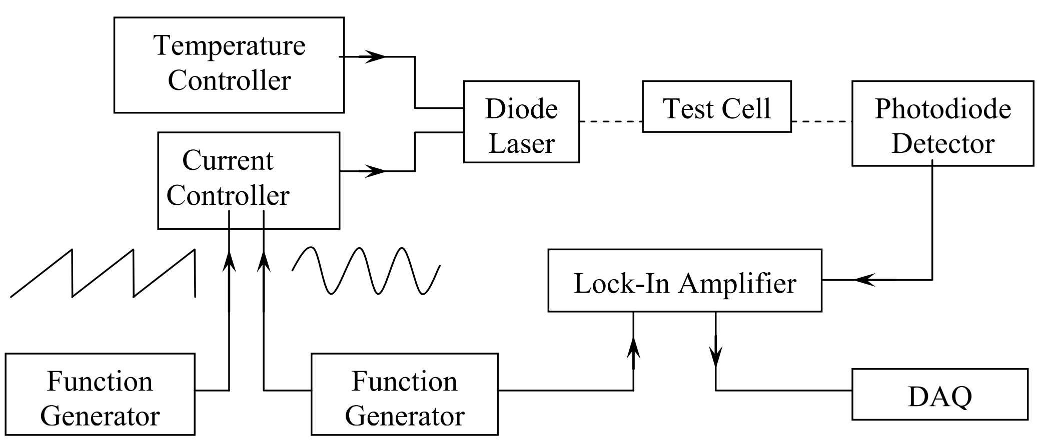

Figure 1: Experimental Setup

In the second period, a sapphire fiber was also tested for its durability in a high temperature, 1900K, environment showing the importance of using sapphire as the material of choice. Evanescence tests were first implemented using the less expensive silica fibers in which geometric changes to the fiber input were experimentally examined. The optimum arrangement found was then replicated on a single 1-m long sapphire fiber which was tested in the high temperature laboratory flame. Modifications to the sapphire fibers were also performed in order to increase evanescent absorption interaction along the fiber. It was found that by using a thin, tapered, optical fiber the detection of evanescent absorption was possible. Tapering the fiber through the use of hydrofluoric acid yielded fiber diameters of about 13 µm for the fiber used and evanescence signal was produced. Evanescence was measured with large CO_2 concentrations and the ability of measurements with small concentrations requires further improvement of the measurement along with other modifications to the sensor.

The experimental setup for the evanescent tests with the tapered fiber is similar to those of previous tests. Signal comparisons between absorption using the lock-in amplifier (harmonic detection) and direct absorption were compared. In this test there was no cell to hold the tapered fiber; instead a CO_2 jet was placed above the tapered region of the fiber allowing for various concentrations of CO_2 to be flow over the taper. This jet located above the fiber can be seen in Figure 2. A jack with a holder for the HF acid is also shown in the figure. This jack allowed for the removal of the acid from the fiber after a suitable taper was created.

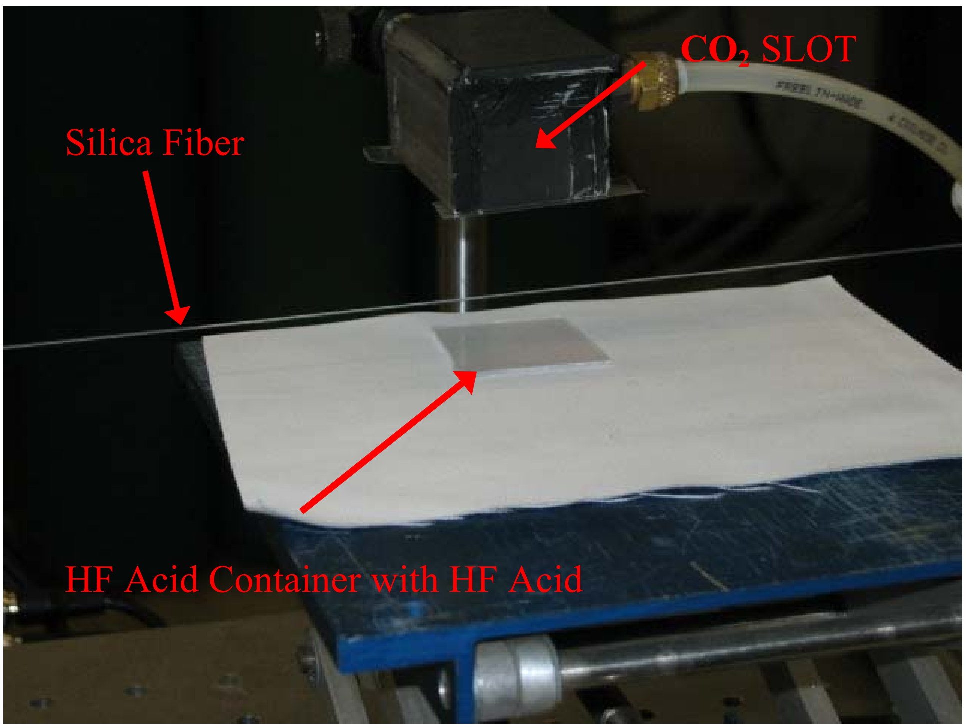

Figure 2: Silica fiber test area, showing acid container and CO_2 jet.

Tapered fibers were successfully created using the method described above. An image of the tape, taken on a SEM microscope, is shown in Fig. 3 where the minimum fiber diameter was about 13 µm. At these diameters the fiber is very susceptible to breakage and other disturbances. During data acquisition it was determined that changes in the flow velocity around the fiber created changes in the signal passing through the fiber as the fiber deformed. To avoid this problem calibration of all flows were performed such that the momentum of the flow passing across the fiber was matched to minimize these effects.

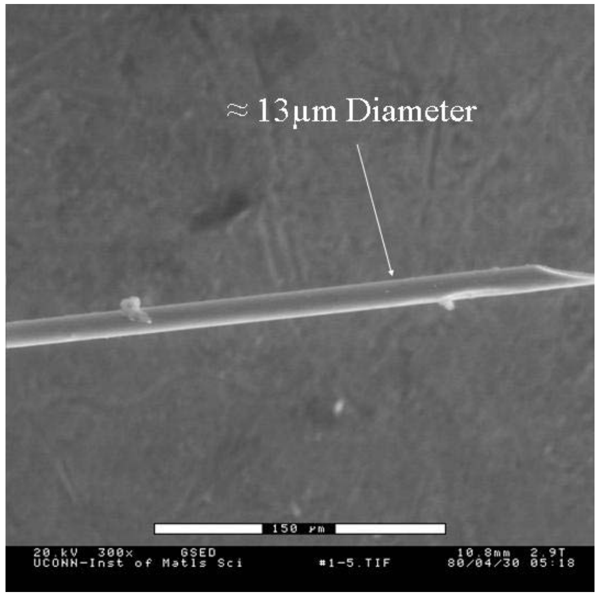

Figure 3: SEM image of a tapered silica fiber.

Transmission though the tapered fiber also created etalon formation within the taper region that was found to be close in magnitude to evanescent absorption. When the wavelength of the laser was varied as done previously, to pass over three CO_2 rovibrational absorption peaks the peaks were hidden by etalon fringe formation. In an attempt to help reduce this, the mean wavelength of the laser was fixed to one absorption peak such that oscillations of the laser wavelength would pass over only one peak. Figure 4 shows a change in signal from the lock-in amplifier when the gas flowing over the fiber was changed between nitrogen and carbon dioxide. Etaloning and noise are still present in these measurements and can be seen in the figure. From the literature it is shown, that a further decrease in the fiber diameter should help with evanescence measurements however the fiber becomes extremely fragile.

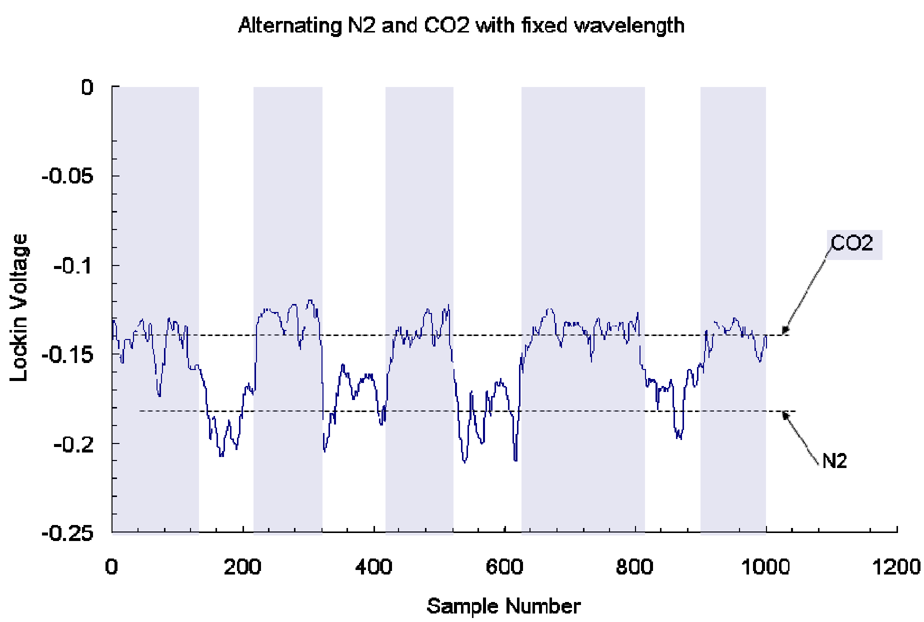

Figure 4: CO_2 absorption using fiber

During this period we developed an opportunity to perform test measurements at a large-scale power plant facility to determine the feasibility and understanding of test conditions for the developed sensor. Alstom power located in Windsor Locks, CT, allowed for the use of an access port to their flue gas stack within a test facility they have, at no cost to the project. Alstom power conducted a safety class in which after completion of the course access to the facility at anytime during testing was allowed. Full access during testing allowed for continuous improvement of the sensor between numerous visits to the facility. Different obstacles, tested designs, and some results from the tests, will be discussed within the results section of this report.

Sensor Development

Setup for in-situ measurements was similar to that of the laboratory experimental setup in Fig. 1; however there was no test cell for measurements, as this was replaced by the flue stack at Alstom Power. The allowed location for measurements was at the flue stack downstream of the scrubbers which yielded relatively clean flow conditions compared to conditions within the boiler. This point was selected as a first step in sensor development and placement, where if the sensor could be created to work in this region it could be further modified in stages until it work in the desired burner location of the facility. The 16" diameter flue stack had a 3 inch port perpendicular to the stack allowing for the connection of any sensor. This region is also optimal for preliminary testing because of relatively low temperatures compared to that of the boiler area or present. Flow at about 180°F passes through the area where the sensor was placed. At these temperatures cheaper materials not required to withstand high temperatures could be used for design of sensor, and optimization. Large flow velocities through the flue stack however provide a challenge with fabrication of a sturdy sensor which will not break, fatigue, and lose alignment.

For initial tests, the use of a fiber would not be possible without breaking the fiber due to the fibers very low strength at diameters needed for evanescence, shown previously. Because of the high flows and particulates within flue gas flows, the fiber implemented into the sensor as a means to retrieve the beam as it passes across the flue gasses would need to be supported to allow for evanescence to take place while having flow still pass along the fiber. The particulates traveling within the flue gas stream attached to the sensor described later as seen in Figure 4.

The first sensor design consisted of two silica rods, through which the absorption beam could enter and exit. Both rods were ground such that one end had a 45° angle on them reflecting the beam out of the rod and then back into the other rod. The silica was able to withstand the temperature within the stack, however alignment was very sensitive and the high temperature flow possibly steered the beam preventing the beam from entering back into the silica rod for measurements.

The next iteration of the design consisted of just silica windows with the beam coming back through the use of a retro reflector held at the end of a metal rod. An image of the retro reflector, attached to the current design can be seen later in Fig. 4. The two small silica windows, constructed from the silica rods allow for the beam to pass through one window toward the retro reflector, and then reflect back to the other window. The laser was fired into one window, while the beam detecting photodiode was placed at the exit of the other window. Alignment of the beam was performed while the sensor was not within the stack. After alignment was complete, the three inch access port was opened and the sensor was carefully inserted into the port. During insertion ,caution was used not to hit the sensor on the inside of the flue stack as large drag forces from flow crossing across the sensor are present. After insertion of the sensor it was noticed that alignment of the laser beam was lost. It was determined that beam steering by the hot gasses across the beam was the cause for this after multiple attempts to carefully place the aligned sensor into the access port without bumping. Due to the beam steering alignment of the sensor would have to be performed after placement into the access port. The original design however, with the two small optical windows did not provide enough flexibility to align the beam after beam steering of the beam.

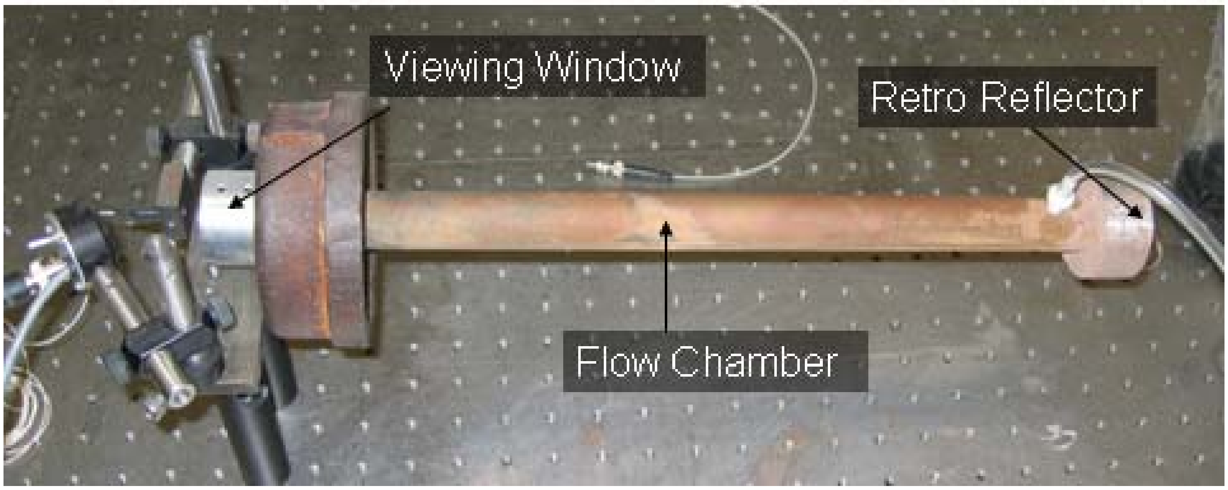

Figure 3: Latest iteration of sensor design for direct absorption measurements within a flue stack.

The next iteration for the design of the sensor involved improving the adjustable alignment range while also minimizing beam steering effects from the flow. Modifications include adding a larger optical access port window at the wall of the sensor. One large window replaced the two small ports initially on the sensor, the window can be seen in Figure 5 along with the mounts for the photodiode and the laser beam collimator. The optical window used also has an antireflective infrared coating on it to help prevent reflections from the surface of the window back to the photodiode. Beam steering occurs when the laser beam passes through regions where the index of refraction of the gas varies across the beam. Gases passing across the beam are at varying temperatures and densities causing this beam steering to take place. To prevent this from taking place the beam was passed within a long tube, with one inlet and an outlet so that gasses from only one flow location pass across the beam. By extracting flue gasses from one location, shown in Fig. 4, temperature gradients from the flow will be minimized and beam steering will be reduced. Figure 3 shows the complete design of the sensor used to make measurements within the flue stack of the Alstom power plant.

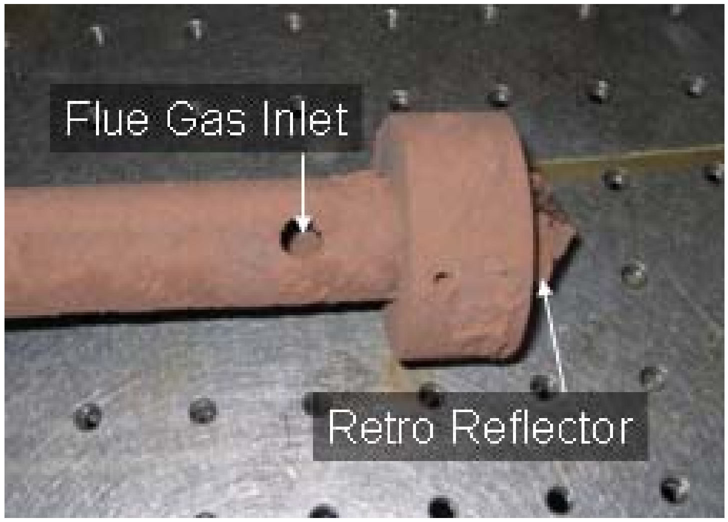

Figure 4: Retro reflector at the end of the flue gas channel, acting to send back the laser beam parallel to itself. Particulate accumulation on the sensor can be seen after several hours of tests at Alstom Power.

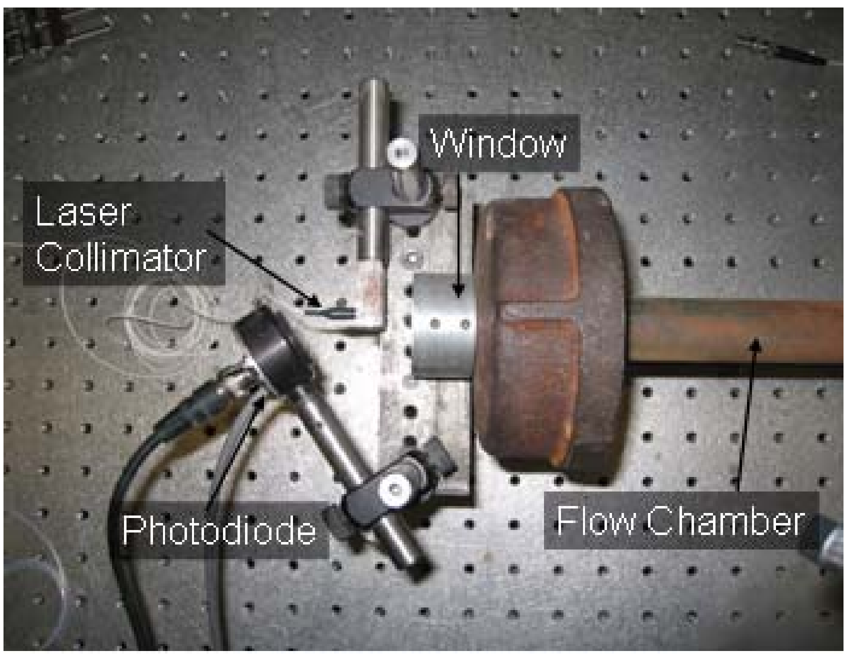

Figure 5: Mounts for alignment of laser beam collimator and photodiode.

Results from Developed Sensor

Measurements using the developed sensor described above within the flue stack at the large scale facility were performed. Using the setup described earlier carbon dioxide absorption spectra are shown in Fig. 6. Three absorption peaks are distinguishable within this range of wavelengths used. Rotation of the sensor 90° caused the flow passing over the sensor to create a low pressure zone at the flue inlet and outlet of the sensor. This lower pressure created a suction of the air outside the sensor filling the flow chamber with air. Measurement of the absorption with air within the chamber was used as a background. The background image with relatively low carbon dioxide concentrations, Fig. 7, was subtracted from the raw CO_2 data to yield background corrected CO_2 absorption spectra Fig. 8. The design of the sensor yielded significant etaloning effects from the retro-reflector and the window used for measurements. Fiber sensors developed in the future could be tuned such that the scale of the etalon can be manipulated by the length of the fiber used in the sensor, the etalon frequency can be made such that its effects will be minimal on the absorption spectra.

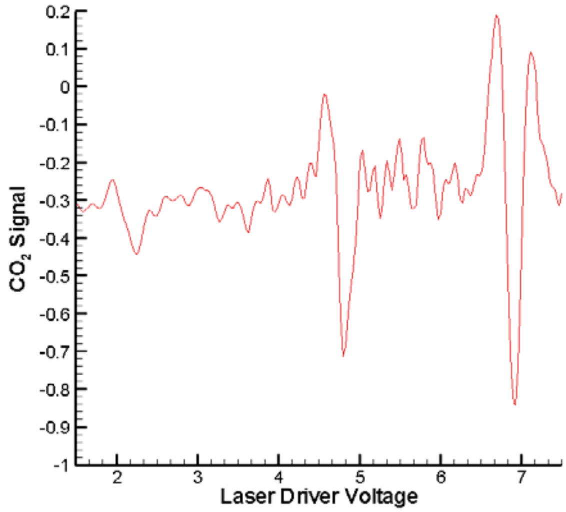

Figure 6: Direct carbon dioxide measurements from developed sensor within flue stack.

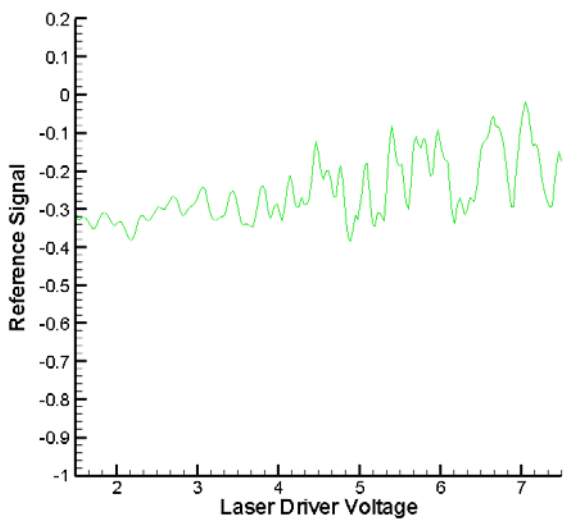

Figure 7: Background signal measured with sensor still within flue stack.

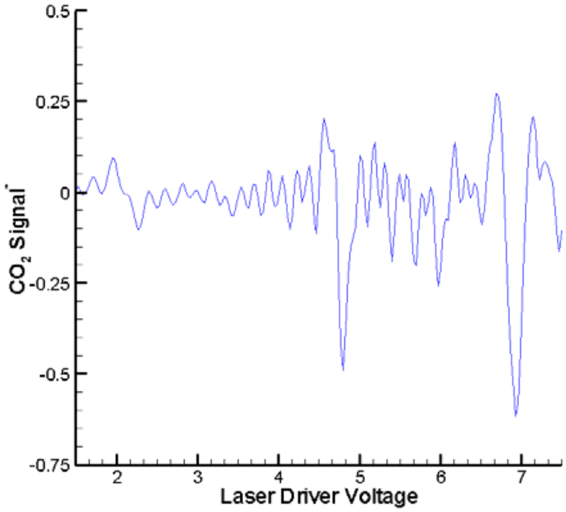

Figure 8: Background corrected CO_2 absorption spectra

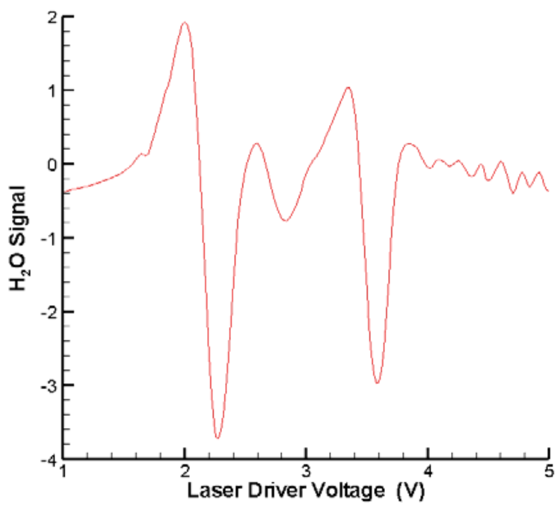

Figure 9: Water vapor absorption spectra within flue stack.

Changing the laser which is connected to the sensor developed allows for the detection of other species within the flow. The detection of water vapor within the flue gasses was measured and can be seen in Figure 9. Two absorption peaks were measured at the wavelength range selected for operation of the laser. The same change of laser to measure other species can be implemented into the evanescent fiber measurements.

Conclusions and Future Work

Evanescent results shown in the last section are very promising, however further work must be performed in an attempt to clean and increase the signal if any significant CO_2 concentration measurements are to be performed. A working sensor for measurements within a flue stack was developed using the direct absorption technique. Knowledge from this sensor, placement and flow conditioning related, can now be applied to development of a fiber sensor which must withstand harsh flow conditions. Some ideas include placing the fiber on a CO_2 (or other desired species) permeable substrate in an attempt to support the fiber under smaller tapered diameters, there in increasing the absorption signal. The ability for the sensor to measure other species, in this case water vapor, was also confirmed and will be applied to the developed sensor.

Currently numerous ideas have been experimented with in order to increase the signal to noise ratio of the evanescence. The sapphire fiber has been tested to withstand temperatures of 1900K showing that it is a suitable material for applications in higher temperature environments. New ideas for increasing the evanescent signal have been determined from the literature. Two methods include tapering the fiber to diameters of around 10 μm and also using a whisper gallery mode which consists of placing a micro-sphere in contact with a tapered fiber such that the light from the laser enters the sphere and reflects internally within the sphere to increase the absorption path length of the laser.

References

1. Ebert, V., Fitzer, J., Gerstenberg, I., Pleban, K.-U., Pitz, H., Wolfrum, J., Jochem, M., and Martin, J., Proc. Combust. Inst. 27:1301 (1998).
2. Deguchi Y, Noda M, Fukuda Y, Ichinose Y, Endo Y, Inada M, Abe Y, Iwasaki S, Meas. Sci. Technol. 13:R103-R115 (2002).
3. Teichert, H., Fernholz, T., and Ebert, V., Appl. Opt. 42:2043 (2003).
4. Lackner, M., Totschnig, G., Winter, F., Maiorov, M.A., Garbuzov, D.Z., Connolly, J.C., Meas. Sci. Technol. 13:1545 (2002).
5. Totschnig, G., Lackner, M., Shau, R., Ortsiefer, M., Rosskopf, J., Amann, M.-C., and Winter, F., Meas. Sci. Technol. 14:472 (2003).
6. Wang, J., Sanders, S.T., Jeffries, J.B., and Hanson, R.K., Appl. Phys. B 72:865 (2001).
7. Schilt, S., Thevenaz, L., and Robert, P., Appl. Opt. 42:6728 (2003).
8. Sonnenfroh, D.M. and Allen, M.G. AIAA Paper 96-2226, 1996.
9. Sonnenfroh, D.M. and Allen, M.G., Appl. Opt., 35:4053 (1996).
10. Zolo Technologies (2004), http://www.zolotech.com/sub/power/zoloboss.php
11. Penner, S.S., Quantitative Molecular Spectroscopy and Gas Emissivities, Addison-Wesley, 1959
12. Harrick, N.J. and Loeb, G.I., Anal. Chem. 45:687-691 (1973).
13. Harrick, N.J., Phys. Rev. Lett. 4:224 (1960).
14. Lee, S.T., Kumar, R.D., Kumar, P.S., Radhakrishnan, P., Vallabhan, C.P.G., and Nampoori, V.P.N., Opt. Comm. 224:237-241 (2003).