On-Line Self-Calibrating Single Crystal Sapphire Optical Sensor Instrumentation for Accurate and Reliable Coal Gasifier Temperature Measurement

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Abstract

This report summarizes technical progress October 2004 – March 2005 on the Phase II program “On-Line Self-Calibrating Single Crystal Sapphire Optical Sensor Instrumentation for Accurate and Reliable Coal Gasifier Temperature Measurement”, funded by the Federal Energy Technology Center of the U.S. Department of Energy, and performed by the Center for Photonics Technology of the Bradley Department of Electrical and Computer Engineering at Virginia Tech.

The outcome of the first phase of this program was the selection of broadband polarimetric differential interferometry (BPDI) for further prototype instrumentation development. This approach is based on the measurement of the optical path difference (OPD) between two orthogonally polarized light beams in a single-crystal sapphire disk. The objective of this program is to bring the BPDI sensor technology, which has already been demonstrated in the laboratory, to a level where the sensor can be deployed in the harsh industrial environments and will become commercially viable.

Due to the difficulties described on the last report, field testing of the BPDI system has not continued to date. However, we have developed an alternative high temperature sensing solution, which is described in this report.

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1.0 Introduction

In the first phase of this program, five different optical temperature sensing schemes were thoroughly investigated to determine an optimal approach for high temperature measurement in coal gasification systems. Based on comparative evaluation and analysis of the experimental results, the broadband polarimetric differential interferometry (BPDI) was chosen for further prototype instrumentation development. This approach is based on the self-calibrating measurement of the optical path difference (OPD), *i.e.* phase retardation between the two orthogonally polarized light beams in a single-crystal sapphire disk, which is a function of both the temperature dependent birefringence and the temperature dependent dimensional sizes.

Due to the difficulties described in our previous report, further testing of the BPDI system has not progressed. This report describes an alternative technology for temperature monitoring in slagging coal gasifiers based on sapphire optical fiber. The principle of the sensor, the interrogating system, the signal processing algorithm and preliminary test results up to 1600°C are described. Issues in the design of sensor packaging and installation for applications in slagging coal gasifiers and the advantages using sapphire as a sensing and protection material are discussed.

2.0 Executive Summary

This report summarizes the technical progress over the fifth six month period of the Phase II program “On-Line Self-Calibrating Single Crystal Sapphire Optical Sensor Instrumentation for Accurate and Reliable Coal Gasifier Temperature Measurement”, funded by the Federal Energy Technology Center of the U.S. Department of Energy, and performed by the Center for Photonics Technology of the Bradley Department of Electrical and Computer Engineering at Virginia Tech.

During the reporting period, research efforts under the program were focused on the following.

- Design and assembly of a new sapphire fiber interferometric sensing system,
- Development of the signal processing algorithm,
- Preliminary testing of the sapphire fiber sensor
- Analysis of the packaging requirements
- Packaging design

EXPERIMENTAL

3.0 Sensing Principle and Structure

Traditional extrinsic Fabry-Perot interferometric (EFPI) sensors usually use two fibers to construct the Fabry-Perot (FP) cavity. For these sensor, it is difficult to generate interference fringes for multi-mode illumination due to their high sensitivity to the surface smoothness, the surface flatness, the distance and the parallelism of the two surfaces of the FP cavity. For highly multi-moded sapphire fibers which are widely used in high-temperature applications, this is even more difficult. Very careful alignment is usually required. Our solution to this problem is to use a sapphire wafer as the interferometer. Since high surface quality and excellent parallelism can be readily achieved in the current wafer lapping/polishing industry, fringes can be easily generated even for highly multi-moded sapphire fiber and large cavity length (thick wafer). The choice of a relatively large thickness is important as it offers great convenience to the signal processing, as described below.

The sensing principle is shown in Figure 3.1. Reflections at both sides of the diaphragm will interfere with each other, producing a modulated spectrum, whose pattern is determined by the optical thickness (OT) of the wafer. The OT is the product of the refractive index and the thickness of the wafer, both of which have thermal dependence, resulting a temperature-sensitive OT and spectrum. Therefore the temperature can be demodulated from the change in the reflected spectrum.

Figure 3.1. Sensing principle.

Figure 3.2. White-light interferometric system setup.

A diagram of the system is shown in Figure 3.2, consisting of a 850nm LED source, a multimode (MM) 3-dB coupler and an OceanOptics S2000 spectrometer. Figure 3.3 shows an enlarged view of the prototype sensor. A 99.8% alumina tube is used as supporting structure, to which both a 59 μm -thick sapphire wafer and a 75 μm (diameter) sapphire fiber are bonded by high temperature alumina adhesive. The sapphire fiber is coupled with a 100/140 μm MM silica fiber which is connected to the system.

Figure 3.3. Diagram of the sensor head.

4.0 Signal Processing

Light from the LED travels through the 3-dB coupler to the sensor head and is reflected. The reflected signal propagates back to the spectrometer, from which the spectrum data is retrieved and processed by a computer. Figure 4.1 shows the spectrum from the sensor head at 25°C and 1597°C, where interference signals are clearly seen. The background intensity is significantly raised by the blackbody radiation at 1597°C. Also, the spectrum is red-shifted, as indicated by the position of the same fringe (interference minima #246) moving from 844.0 nm at 25 °C to 869.7 nm at 1597 °C.

Figure 4.1. Sensor spectrum at different temperatures.

In general, the total light intensity received at the spectrometer can be written as

$$\begin{aligned}
I_t(\lambda) &= I_B(\lambda) + I_D(\lambda) + I_{LED}(\lambda)(r_{couple} + r_{end} + r_{s1} + r_{s2} - 2\sqrt{r_{s1}r_{s2}} \cos \frac{4n(T)d(T)\pi}{\lambda}) \\
&= I_B(\lambda) + I_D(\lambda) + I_{LED}(\lambda)(r_{couple} + r_{end} + r_{s1} + r_{s2}) - 2I_{LED}(\lambda)\sqrt{r_{s1}r_{s2}} \cos \frac{4n(T)d(T)\pi}{\lambda}, \quad (4-1)
\end{aligned}$$

where $I_B(\lambda)$, $I_D(\lambda)$ and $I_{LED}(\lambda)$ is the light intensity of blackbody radiation, dark current and LED. r_{couple} , r_{end} , r_{s1} and r_{s2} are reflections at every surfaces, the coupling point of silica fiber and sapphire fiber, the end face of the sapphire fiber, the first and second surfaces of the sapphire wafer, respectively. n is the refractive index of sapphire and d is the thickness of the wafer. Both of them are temperature-dependent. In Eq. (4-1), the first three terms represent the light intensity from blackbody radiation, dark current and reflections at every surfaces, all of which are slow-varying and thus far will be located close to the zero frequency after a Fourier transform of the spectrum. On the other hand, the last item, which contains the cosine, is a high frequency component and will be separated from the first three items by Fourier transform. A band-pass filter can be used to filter out all the low frequency components, leaving only the last item modulated by the LED spectrum profile $I_{LED}(\lambda)$. This profile can be extracted by a Hilbert transform using the techniques of envelope detection as the following

$$\begin{aligned}
\hat{x} &= H(x) = 2I_{LED}(\lambda)\sqrt{r_{s1}r_{s2}} \sin \frac{4n(T)d(T)\pi}{\lambda} \\
I_{env} &= (x^2 + \hat{x}^2)^{1/2} = 2I_{LED}(\lambda)\sqrt{r_{s1}r_{s2}} \cos \frac{4n(T)d(T)\pi}{\lambda} \quad (4-2)
\end{aligned}$$

where x denotes the last item in Eq. (4-1) and $H(\cdot)$ denotes the Hilbert transform. Therefore normalization of x by I_{env} leads to a pure cosine term, $\cos \frac{4n(T)d(T)\pi}{\lambda}$, which corresponds only to the parameters of the sapphire wafer, namely its refractive index and thickness. By tracing the change of this term when temperature varies, a dependence of $n(T)d(T)$ on T can be determined and used for temperature demodulation.

Figure 4.1 shows the Fourier transform of the spectrum. The side peaks (high frequency component) are the interference signal and the central peak (low frequency component) consists of blackbody radiation, CCD dark current, LED spectrum profile and other reflections along the light path. The interference signal is extracted by filtering the central peak out using a band-pass filter and then normalized, as shown in Figure 4.3. This spectrum is related to the optical thickness (OT) of the sapphire wafer or optical path difference (OPD) between two reflections at the surfaces as in Eq. (4-1). The negative sign is due to the additional phase change of π due to the reflection at air-sapphire interface.

$$I(\lambda) = -\cos \frac{4n(T)d(T)\pi}{\lambda} = -\cos \frac{4\pi OT}{\lambda} = -\cos \frac{2OPD\pi}{\lambda} \quad (4-3)$$

Figure 4.2. Fourier transform of the spectrum.

Figure 4.3. Spectrum of filtered high frequency component.

Figure 4.4. Filtered and normalized spectrum.

5.0 Test Results

The sensor was placed in a high temperature furnace along with a B-type thermocouple, the tip of which is next to the sapphire wafer. The furnace was cycled from about 230°C to 1600°C three times. The OT change with respect to temperature measured by B-type thermocouple is plotted in Figure 5.1. A calibration curve was obtained by fitting all the data using a 4th order polynomial. This curve converts an OT value from the sensor to a temperature value, which is then taken as the temperature output of the sensor. Figure 5.2 shows the temperatures from both the sensor and the thermocouple after the conversion. Figure 5.3 plots their difference, which varies from -3°C to 3°C, corresponding to a $\pm 0.2\%$ full scale accuracy. This may be largely due to the $\pm 0.5\%$ FS accuracy of the thermocouple.

Figure 5.4 shows a system resolution test at room temperature. The sensor was kept in the furnace at room temperature and optical thickness was continuously recorded for 5 minutes. The standard deviation σ is about 0.33 nm. The sensitivity of the sensor at room temperature is about 1.46 nm/°C. So the 2σ -resolution is about $2 \times 0.33 \text{ nm} / 1.46 \text{ nm/°C} = 0.45 \text{ °C}$. Instead of processing only one spectrum at a time, recording additional spectra and taking the average can reduce the noise and further improve resolution. A four-spectra average can reduce the standard deviation to 0.165 nm, which is $1/2$ ($1/\sqrt{4}$) of the result without averaging. Increasing the averaged spectra to 9 will further reduce this value to 0.105 nm, corresponding to $1/3$ ($1/\sqrt{9}$) of no averaging and about 0.15°C temperature resolution.

Figure 5.1. OT vs. temperature for three thermal cycles.

Figure 5.2. Temperature: sensor vs. thermocouple.

Figure 5.3. Temperature difference between sensor and thermocouple.

Figure 5.5 plots the standard deviation of 5-minute data for different numbers of averaged spectra. The x-axis is plotted as the square root of the number of averages. Therefore, the y-axis would be expected to be inversely proportional to x-axis if the variation of the optical thickness is white-noise-like. As we have already seen, the first three points follow this relation very well. However, for additional averaging, the standard deviation does not reduce further but reaches a limit. The temperature variation inside the furnace should account for this limit. An environment with better controlled temperature will reduce this variation limit and reveal the improved resolution by averaging a greater number of spectra.

The CCD integration time for each spectrum is 20ms. Even for a 50-spectra average, the data acquisition is still about 1Hz, which is sufficient for most temperature applications. Besides taking averages, since sensitivity increases significantly with temperature, the resolution is also expected to be enhanced for high temperature measurement.

Figure 5.4. System resolution test.

Figure 5.5. System resolution vs. number of averages.

RESULT AND DISCUSSION

6.0 Sensor Probe Packaging Design

6.1 Packaging Requirements

The optical sapphire temperature sensor developed is intended for direct high temperature measurement in the primary and secondary stages of slagging gasifiers. Several engineering issues have to be resolved at the prototype development stage, including the following.

High temperature capability — The operating temperature in the coal gasifier is in the range of 1200°C~1600°C, depending on the physical location in the chamber. To measure high temperatures accurately in a wide measurement range with high resolution, proper fabrication materials are needed.

High pressure capability — Background pressure as high as 500psig can be encountered in the coal gasifier chamber. The sensor optical elements should be able to withstand such environmental pressure. The sensor enclosure should meet the industrial safety requirements, while not conflicting with sensor optical architecture.

Chemical corrosion resistance — With temperatures exceeding 1200°C, pressure exceeding 500psig, and chemically corrosive agents such as alkalis, sulfur, transition metals and steam, proper fabrication materials are needed to implement the sensing probe

Deployable — Optical temperature sensors designed for harsh environment applications must be capable of remote operation and be flexible enough for easy deployment. Features of mechanical vibration-proof, high mechanical strength, and remote monitoring and control capability are thus necessary.

6.2 Sensor Probe Packaging Design Example

This section describes a packaging example designed to meet all these requirements, as shown in Figure 6.1. The sensor is mounted to the coal gasifier shell through a mounting flange. Functionally, the sensor is divided into three sections: the probe, the first and second pressure isolation chamber. The first pressure isolation chamber sees coal gasifier operation pressure when coal gasifier is in operation. The second pressure isolation chamber sees room pressure when first pressure isolation chamber is working. The pressure boundary of the first pressure isolation chamber is the flange connection on the mounting nozzle, the flange connection with the second pressure isolation chamber and the fiber feedthrough. The pressure boundary of the second pressure isolation chamber is the flange connection with the first pressure isolation chamber and the fiber feedthrough. In case first pressure isolation chamber fails to bound the pressure, the second pressure isolation chamber will take the role as the pressure boundary, therefore prevent the hot air from shooting out from the sensor mounting nozzle. The double pressure isolation design minimizes the chance of accident during coal gasifier operation.

Figure 6.1. Overall sensor probe packaging design.

A detailed view of the second pressure isolation chamber is shown in Figure 6.2. The chamber material could be carbon steel or stainless steel. The wall thickness should allow the chamber to withstand the sudden coal gasifier operation pressure impact. The gasket used in the flange connection should be graphite, because during the coal gasifier operation, when the heat transfer is balanced, the pressure isolation chambers see temperature that is much higher than room temperature. The choice of graphite is to prevent the gasket from failure under high temperature. The flange-type fiber feedthrough is through a multimode fiber to the sensor electronics.

Figure 6.2. Second pressure isolation chamber.

The two fiber feedthroughs used in the pressure isolation chamber are flange-type. The fiber feedthroughs are used to guide the light power to and from the sensing probe. The fibers are connected together through fiber connectors and adaptors, which could be SMA, FC or ST types. The fiber feedthrough can be purchased from a number of vendors such as: Fiberguide, OZ Optics LTD, Ceram Optee, Johonson Hermetic Feedthrough, etc.

A detailed view of the sensor probe is shown in Figure 6.3 and Figure 6.4. The sensor is enclosed in a closed-end inner alumina tube. The alumina spacers are attached to it to match the diameter of the inner and outer alumina tube. This is to minimize impact-induced damage during installation and vibration during operation. The sensor protected by the inner alumina tube is inserted into an 18" long one-end-closed sapphire tube. The entire sapphire tube is made of single-crystal sapphire and therefore can withstand high temperature and aggressive corrosion. The inner alumina tube and the sapphire tube are enclosed by an outer 99.8% alumina tube to provide support as well as corrosion protection to the sensor. These tubes are bonded together through high temperature ceramic glue that has a thermal expansion coefficient similar to alumina and sapphire. Thus, the sensor has three layers of corrosion protection: high purity alumina—single crystal sapphire—high purity alumina, and this structure is believed capable of corrosion protection in coal gasifier environment. The outer alumina tube is bonded to a stainless steel tube that provides support and protection. An aluminum pipe is connected with the stainless steel tube via a reduced coupler to provide extra protection to the sensor head during installation. The sensor probe is ended with a fiber connector that connects with the fiber feedthrough of the first pressure isolation chamber.

A close up of the first pressure isolation chamber is shown in Figure 6.5. The sensor probe is fixed to the pressure isolation chamber by two reduced couplers. The two reduced couplers give support to the weight of the sensor probe and prevent tilting and vibration. The fiber feedthrough and sensor probe connection is in the first pressure isolation chamber. It could be either the fiber connector type or splice connection type. The flange-type fiber feedthrough is fixed onto the chamber with a graphite gasket in between.

Figure 6.3. Probe design.

Figure 6.4. Close up of sensor head.

Figure 6.5. Close up of first pressure isolation chamber.

7.0 Use of Sapphire in Harsh Environments

In the previous discussions, sapphire was used for both the sensing elements (fiber and wafer) and the packaging material (protection tube). This section will discuss the advantages of using sapphire in high-temperature harsh environments, based on corrosion data of sapphire tested in coal slag.

Single crystal sapphire has unique physical, chemical and optical properties such as high melting temperature, high hardness, relatively good optical transmission windows, and optical birefringence. The combination of these properties may allow sapphire to withstand the high temperature, high pressure, thermal shock, abrasion and erosion in harsh environments, such as those found in commercial gasifiers and glass melting operations. Single crystal sapphire is chemically composed of pure aluminum oxide, Al_2O_3 . Various high purity polycrystalline aluminas are also available. Sapphire has a hexagonal/rhombohedral crystal structure which determines a number of properties exhibited by sapphire. The main advantage of single crystal sapphire over polycrystalline alumina is that it is non-porous and contains no grain boundaries. Therefore use of single crystal sapphire will eliminate the effect of corrosion along the grain boundaries. The melting point of sapphire is $2053^{\circ}C$ and it is able to withstand extreme thermal shock conditions due to its high strength and high thermal conductivity. Sapphire is an attractive optical material. Its large energy gap of 9.1eV enables it to have superior optical transmission compared to other oxide materials. In addition, sapphire has the ability to transmit at extremely high temperature. Sapphire has exceptionally high chemical stability even at high temperatures in oxidizing environments. It is the hardest oxide material. It's hardness of 9 on Mohs scale, closely compares to that of diamond, which is 10.

The table below shows the thickness change of sapphire sample exposed to coal slag at the temperatures and times noted. As shown in the data, the resistance of single crystal sapphire to corrosion by the coal slag is quite high even at very high temperatures.

Table 7.1. Corrosion of sapphire in coal slag at $1200^{\circ}C$, $1300^{\circ}C$ and $1400^{\circ}C$ over 4 days.

Days in furnace	HF method			SEM method		
	$1200^{\circ}C$	$1300^{\circ}C$	$1400^{\circ}C$	$1200^{\circ}C$	$1300^{\circ}C$	$1400^{\circ}C$
1	-0.01	0.01	0.02	-0.0827	0.0615	
2	-0.01	-0.01	0.05	0.0387	0.0427	
3	0	-0.02	0.04	0.0476	0.0385	
4	0.01	-0.01		-0.0505	-0.0497	-0.1025

*Data in columns 2 and 3 were initially reported in our report issued November 2003

CONCLUSIONS

8.0 Conclusions and Future Work

The main objective of this project is to bring the sensor technology, which has already been demonstrated in the laboratory, to a level where the sensor can be deployed in the harsh industrial environments and will become commercially viable. Due to numerous logistical problems, data was not able to be retrieved from the BPDI sensor system at the field test site. Research efforts during the reporting period were thus focused on development of an alternative sensor system utilizing Fabry-Perot interferometry. A new optical structure and signal processing algorithm have been developed. Preliminary experimental results were presented. Following an analysis of the requirements, a sample packaging design was developed. Overall, the data indicate that the single crystal sapphire elements should exhibit good corrosion resistance when subjected to coal slags at high temperatures such as those found in coal gasification systems.

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List of Acronyms and Abbreviations

A/D, analog to digital
APP, Advanced Pressure Products, Inc.
BPDI, broadband polarimetric differential interferometry
CCD, charge couple device
CPT, Center for Photonics Technology
CTE, coefficient of thermal expansion
EFPI, extrinsic Fabry-Perot interferometer
EMI, electromagnetic interference
FP, Fabry-Perot
FWHM, full width half maximum
GPM, gallons per minute
GRIN, graded index
ID, inner diameter
LED, light emitting diode
MM, multimode
MMF, multimode fiber
OD, outer diameter
OPD, optical path difference
OT, optical thickness
PC, personal computer
PZT, lead zirconium titanate
RFLWN, raised face long weld neck
SCIIB, self-calibrated interferometric/intensity-based
SLED, superluminescent light emitting diode
SMF, single mode fiber
SNR, signal to noise ratio
VTPL, Virginia Tech Photonics Laboratory (now Center for Photonics Technology)