Smart Structures for Application in Ceramic Barrier Filter Technology

**Final Report
August 1991 - August 1994**

S.J. Weinstein
T.E. Lippert

December 1994

Work Performed Under Contract No.: DE-AC21-91MC28020

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Westinghouse Electric Corporation
Pittsburgh, Pennsylvania

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FILTER TECHNOLOGY

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Westinghouse Electric Corporation
Science and Technology Center
1310 Beulah Road
Pittsburgh, Pennsylvania 15235-5098

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ABSTRACT

High temperature optical fiber sensors were developed to measure the in-service stressing that occurs in ceramic barrier filter systems. The optical fiber sensors were based on improvements to the sensor design developed under the DOE/METC Smart Structures for Fossil Energy Applications contract no. DE-AC21-89MC25159. In-house application testing of these sensors on both candle and cross-flow filters were performed in the Westinghouse Science and Technology Center High-Temperature, High-Pressure Filter Test Facility and the results analyzed. This report summarizes the sensor developments, methods to apply the sensors to the filters for in-situ testing, and the test results from the four in-house tests that were performed.

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1. INTRODUCTION

The Westinghouse Science and Technology Center (STC) is currently developing cross flow and candle filter systems for advanced fossil power generation application. Current filter development efforts have utilized mathematical models to predict in-service stressing of filters to improve filter life and reliability. These models depend on estimating certain heat transfer and physical property parameters that are not well defined. There is a need to substantiate and refine this analysis with in-situ sensors. Optical fiber sensors have shown to be the only available technology capable of measuring strain and stress in the high-temperature flyash environment of a boiler. The purpose of this program is to utilize improved, high temperature, optical fiber sensors to measure the in-service stressing that occurs in ceramic barrier filter systems, thereby providing valuable input to the optimization of filter processing and operation.

The program was structured as a Base Program of six tasks with an optional three tasks addressing field applications. In the Base Program, STC submitted environmental information required by DOE to complete the appropriate level of National Environmental Policy Act documentation (Task 1). Next, the original optical fiber sensor developer, the Virginia Polytechnic Institute and State University (Va Tech), developed second-generation fiber sensor prototypes, and STC transferred the technology from Va Tech to the STC (Task 2) and optimally applied it to ceramic barrier filters (Task 3). An experimental test plan was made for in-house application testing of the smart filters at STC's HTHP Filter Test Facility (Task 4). This was followed by the application testing (Task 5) and an evaluation and assessment of the tests (Task 6). Two additional tasks were added to the Base Program once it was recognized that the ruggedness of the optical fiber sensors needed improvement. The first task addressed sensor lead fiber breakage and debonding of the sensor assembly from the filter (Task A1). Westinghouse then performed an in-house simulation test and submit recommendations for field tests of the sensors on filters (Task A2).

The Optional Program was to begin by the decision of the DOE based on the results reported in the draft topical report. STC would draft a test plan for field tests at the appropriate site (Task 7), conduct the tests (Task 8), and evaluate and assess the results of the tests with a final report (Task 9).

This final report will address the accomplishments of the Base Program by Task. In the interest of document flow, Tasks 4-6 have been combined on a test-to-test basis.

For clarification, the term, "Smart Structure," refers to the union of sensors with a structure, such that the sensors can monitor the surrounding environment, structural integrity, or other relevant parameters. Ideally, the structure will then be "smart" enough to process the data and execute the necessary steps to optimize its performance efficiency. In this Base Program, optical fiber sensors have been bonded to ceramic barrier filters to show the capability of surviving and sensing strain and stress in simulated boiler conditions.

2. SENSOR IMPROVEMENTS/TECHNOLOGY TRANSFER

2.1 OPTICAL FIBER SENSOR BACKGROUND

The optical fiber sensors used for the tests are based on the design developed under the DOE/METC Smart Structures for Fossil Energy Applications contract (contract no. DE-AC21-89MC25159) by Va Tech. A detailed description of this sensor and its performance on cross flow filters can be found in the Final Technical Report for that contract dated December 18, 1990. Briefly, the sensor is commonly known as an extrinsic Fabry-Perot interferometer (EFPI). An interferometer measures the amount of phase shift between two coherent optical beams by means of optical interference. Typically, a laser beam is split into two beams, one of which is perturbed by the desired parameter and becomes phase-shifted with respect to the other beam. The beams are then recombined and interfere with each other. A detector detects this phase change as a sinusoidal function of light intensity. In the case of an EFPI, one of the interfering beams (the reference beam) is formed by an internal reflection at the end of a cleaved fiber. Of the remaining light that exits the fiber, a portion is reflected back into the fiber (sensing beam) by the cleaved (outer) surface of a second fiber piece, serving as a mirror. The extra distance the sensing beam travels causes the phase delay with respect to the reference beam. This can be seen in Figure 2-1. A hollow tube sleeve (hollow-core fiber) is placed over the sensor for protection and alignment and can be fused to the second fiber to make a more integrated unit. Therefore, if the sensor is attached to a material under test, with attachment points both at the sleeve and at the primary fiber close to the sleeve, an expansion (or contraction) of the material will cause a change in the gap between the primary fiber and the second fiber piece. This gap, in turn, will be transduced into a modulation of the interference pattern of the returning optical signal. The sinusoidal optical signal output (intensity) will have a period on the order of half the optical wavelength used. This means that the sensitivity of gap change can be a fraction of the wavelength of the light. For an EFPI, this sensitivity can be on the order of one nanometer or smaller. Because the source of modulation takes place outside of the primary optical fiber, which serves as a lightpipe to carry the light to and from this region, it is labeled an extrinsic sensor.

The mathematical analysis of the EFPI performance considers the interference of a two-wave interferometer in terms of a plane-wave approximation. A coherent, approximately plane wave detected at the output of the sensor can be represented in terms of its complex amplitude $U_i(x,z,t)$, given by

$$U_i(x,z,t) = A_i \exp(j\phi_i), i=1,2 \quad (1)$$

where the variable A_i can be a function of transverse coordinate x and the distance traveled z , and the subscripts $i=1,2$ stand for the reference and the

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Figure -- 2-1 - EFPI Sensor.

sensing reflections, respectively. Assuming the reference reflection coefficient $A_1 = A$, the sensing reflection coefficient A_2 can be written as

$$A_2 = A \left[\frac{ta}{a + 2s \tan(\sin^{-1}(NA))} \right] \quad (2)$$

where 'a' is the fiber core radius, 't' is the transmission coefficient of the air/glass interface (≈ 0.96), 's' is the gap separation and 'NA' is the numerical aperture of the single mode fiber given by $NA = (n_1^2 - n_2^2)^{1/2}$. The terms n_1 and n_2 are the refractive indices of the core and cladding respectively. The observed intensity of detector is given by

$$I_{det} = |U_1^2 + U_2^2|^2 = A_1^2 + A_2^2 + 2A_1A_2 \cos(\phi_1 - \phi_2) \quad (3)$$

which can be written as

$$I_{det} = A^2 \left| 1 + \frac{2ta}{a + 2s \tan(\sin^{-1}(NA))} \cos\left(\frac{4\pi s}{\lambda}\right) + \left(\frac{ta}{a + 2s \tan(\sin^{-1}(NA))} \right)^2 \right| \quad (4)$$

We have assumed that $\phi_1=0$ and $\phi_2=2s(2\pi/\lambda)$, and λ is the wavelength of operation in free space. Note that we have ignored effect of multiple reflections. For a displacement sensor it is useful to plot the detected intensity versus gap-separation 's', as shown in Figure 2-2a. We see that the fringe contrast drops as the displacement increases, which is to be expected since the relative intensity of the sensing reflection starts dropping with respect to the reference reflection.

The number of fringes that the sensor attached to the filter undergoes due to thermally induced strain can be predicted to be

$$\# \text{ of fringes} = \frac{2l(\Delta T)\alpha_{filter}}{\lambda} \quad (5)$$

where 'l' is the gage length of the sensor, ' ΔT ' is the change in the temperature, α_{filter} is the thermal expansion coefficient of the filter ($4.3 \times 10^{-6} / ^\circ\text{C}$) and ' λ ' is the wavelength of light (1300 nm). To test the calibration of the sensor, a micropositioner was used to create a known displacement between the two fiber ends that form the Fabry-Perot cavity. For a comparison with the theoretical results, an oscilloscope trace of the continuously monitored output intensity of the sensor for a starting gap of zero to a final gap of 203 microns is shown in Figure 2-2b. The experimentally counted number of fringes for a displacement of 202 microns was 301.5. From a theoretical calculation, the gap for this was 312.3, or approximately 3% difference.

The sensitivity of strain measurement depends on the gauge length and the minimum displacement resolution of the sensor. By either using a shorter wavelength of light or increasing the gauge length, or both, one will increase the strain sensitivity. The increase in sensitivity, however, will cause the occurrence of more 360° phase cycles, or interference fringes, for a given strain. In order to monitor large strains with high sensitivity, therefore, the sensor signal processing will need to keep track of many 360° phase cycles.

This cyclic output also introduces a difficulty in monitoring the direction of the strain. To overcome this, a dual interferometer approach is introduced where the outputs are 90° (quadrature) phase-shifted from each other. As expansion occurs, one signal will then always lead the other; when compression occurs, the other signal will lead. The two methods considered to achieve the

(a) Theoretical plot of sensor output fringes

(b) Oscilloscope trace of observed sensor output fringes for increasing gap displacement

Figure 2-2 - Theoretical plot and actual traces of EFPI sensor output. The total displacement of the sensor in (b) was 202 μm , measured at the 1 microstrain level or better. The output is in good agreement with theory and has been shown to have good repeatability.

quadrature phase shift are 1) to multiplex two light sources of different wavelengths into one fiber sensor or 2) to put two sensors side-by-side and bias the phase of the output of one of the sensors by 90° with respect to the other. The first technique is based on the observation that the period of sinusoidal output is directly related to the optical wavelength of the source. A longer wavelength will produce a longer period. As one increases the gap of the sensor, therefore, one can find a region where the output phases of two appropriate wavelengths will be separated by 90° . This quadrature works only within a certain region. At zero gap separation the two signals are completely in-phase, and after a certain separation, the relative phase increases until it reaches 360° , at which point the two signals are back in-phase with each other. Due to the limited range of operation, we decided to use the second technique.

The second technique uses two identical sensors in close proximity to each other. During calibration, the gap of one of the sensors would be changed and fixed at a position where its output is in quadrature with the other sensor. These signals will always be in quadrature unless something upsets only one of the two channels, which is not expected. Figure 2-3 shows two conceptual designs of this second technique. The simulation tests used Version B because of the ease of manufacture.

Typical instrumentation for the sensors is in two stages. The first stage consists of a laser, laser driver electronics, a detector, and output signal pre-amplification stage. The analog signal from the pre-amplification stage is then bused to the next stage, either a digital memory oscilloscope or an analog-to-digital convertor connected to an electronic processing unit. With an oscilloscope, the data can be viewed at the time of the test and remain in memory for post-processing on a computer. The electronic processing unit, on the other hand, can quickly collect and process the signals in real time.

The speed of response of the EFPI sensor family depends on the speed of response of the material to which the sensor is bonded and the speed of response of the signal processing.

2.2 SENSOR IMPROVEMENTS

A number of improvements have been made to the performance of ceramic barrier filter sensor technology developed under the previous DOE smart structures contract. As a subcontractor, Va Tech held the responsibility of the sensor research and development, which included the following items: 1) improvements in the signal-to-noise ratio SNR of the strain sensors, 2) research into a sensor to monitor 3-dimensional strain, 3) analysis of an anomaly in sensor response that occurs at 500°C , 4) application of metal coatings directly on the optical fibers, 5) increases in the response rate of the sensors, 6) development

Figure 2-3 - Conceptual Designs of the Side-By-Side Version of the EFPI Quadrature Phased-Shifted Sensor.

of quadrature phase-shifted sensors for expansion vs. contraction information, in particular a dual-wavelength sensor, 7) development of a method to create an absolute strain sensor that does not require the continuous monitoring of the fringe count, and 8) development of EFPI temperature sensors.

2.2.1 Improvements to the Signal-to-Noise Ratio

2.2.1.1 System Optimization.

The Signal-to-Noise Ratio (SNR) determines the resolution of an EFPI fiber sensor system. For an interferometric fiber sensor, the SNR is related to a number of factors such as the source power level, fringe contrast in the sensor output, optical losses in the system, and signal processing system. In the previous EFPI sensor systems, the SNR was on the order of 15 dB, which corresponded to a resolution of 10 nm in the systems used. In order to improve the resolution, we concentrated on the improvement of the factors mentioned above. Incorporated into the new sensor system were 1) a powerful laser with 10 milliwatts output, 2) 2x2 fiber couplers with low insertion losses, 3) quality fiber splice tubes, and 4) low noise/drift preamplifiers. In doing so, the SNR of the new system has been improved to 35 dB, yielding a resolution of 0.1 nm. In addition, since the low drift preamplifiers were incorporated, no long-term drifts in the sensor output were observed.

2.2.1.2 Microlensed EFPI Sensors.

The peak-to-peak output signal level, or fringe contrast, of the EFPI decreases as the gap separation, s , between the primary fiber and the target fiber piece increases. This is due to the fact that the light diverges as it exits the primary fiber. This causes less light from the sensing beam to re-enter the primary fiber at greater gap separations, while the reference beam remains the same. By collimating the light beam that exits the primary fiber, we can maintain the intensity of the sensing beam, thus improving the dynamic range and SNR of the sensor. This could be done by forming a spherical endface, or microlens on the tip of the primary fiber. It is predicted that the correct radius of curvature of the fiber endface will increase the dynamic range of the sensor approximately five times¹.

The theoretical analysis of the microlensed EFPI sensor was completed in April 1992. The final formulation for the intensity field of light at any point outside the primary fiber is given by

$$I(P) = [C(P)]^2 + [D(P)]^2, \quad (6)$$

¹A. Wang, M.S. Miller, K.A. Murphy, R.O. Claus, "Extrinsic Fabry-Perot interferometric optical fiber sensor with micro-lens", Fiber Optic Sensor-Based Smart Materials and Structures, April 1992.

where

$$\begin{aligned}
 C(P) = & \frac{-1}{4\rho} \iint \left[\frac{1}{S} \cos(kS) J_0(\kappa_{01}\rho) \frac{\partial S}{\partial n} \left(-\frac{1}{S} \cos(\beta_{01}z) \right) \right. \\
 & - \left(\frac{\cos(kS)}{r} \right) \left(\kappa_{01}\rho J_1(\kappa_{01}\rho) \cos(\beta_{01}z) + kz J_0(\kappa_{01}\rho) \sin(\beta_{01}z) \right) \\
 & - \sin(kS) J_0(\kappa_{01}\rho) \frac{\partial S}{\partial n} \left(k \cos(\beta_{01}z) - \frac{1}{S} \cos(\beta_{01}z) \right) \\
 & \left. - \left(\frac{\sin(kS)}{r} \right) \left(kz J_0(\kappa_{01}\rho) \cos(\beta_{01}z) - \kappa_{01}\rho J_1(\kappa_{01}\rho) \sin(\beta_{01}z) \right) \right] ds,
 \end{aligned} \tag{7}$$

and

$$\begin{aligned}
 D(P) = & \frac{-1}{4\rho} \iint \left[\frac{1}{S} \sin(kS) J_0(\kappa_{01}\rho) \frac{\partial S}{\partial n} \left(-\frac{1}{S} \cos(\beta_{01}z) - k \sin(\beta_{01}z) \right) \right. \\
 & - \left(\frac{\sin(kS)}{r} \right) \left(-\kappa_{01}\rho J_1(\kappa_{01}\rho) \sin(\beta_{01}z) + kz J_0(\kappa_{01}\rho) \cos(\beta_{01}z) \right) \\
 & - \cos(kS) J_0(\kappa_{01}\rho) \frac{\partial S}{\partial n} \left(k \cos(\beta_{01}z) - \frac{1}{S} \cos(\beta_{01}z) \right) \\
 & \left. - \left(\frac{\cos(kS)}{r} \right) \left(kz J_0(\kappa_{01}\rho) \cos(\beta_{01}z) - \kappa_{01}\rho J_1(\kappa_{01}\rho) \sin(\beta_{01}z) \right) \right] ds.
 \end{aligned} \tag{8}$$

For the above equations, $\kappa_{01}^2 = \omega^2 n_1^2 / c^2 - \beta_{01}^2$, ω is the angular frequency of operation, β_{01} is the longitudinal propagation constant of the mode, k is the free space propagation constant given by $k = 2\pi/\lambda$, and the integral is evaluated over the core region of the fiber endface. The factor S is the distance between a point Q at the primary fiber endface and the point "P" (Figure 2-4) and can be written as

$$S = \sqrt{\rho^2 + \rho'^2 - 2\rho\rho' \cos(\phi - \phi') + (z' - z)^2}, \tag{9}$$

Figure 2-4 - The Fiber Endface with a Micro-Lens.

where ρ , ϕ , and z are the cylindrical coordinates at the fiber endface and ρ' , ϕ' , and z' are the cylindrical coordinates of the point 'P'. The equations (8) and (9) were calculated and the results are plotted in Figure 2-5. When the light out of the primary fiber is well collimated by the microlens, the total power will concentrate on a small region in the far-field. based on this understanding, the radius of the microlens should be chosen to be approximately 90 μm .

The intensity given above is added to the reflection from the primary fiber end surface, and the result is integrated over the core region of the fiber end surface. This result is the intensity of the light returning back down the fiber to the detector.

Figure 2-5 - Far Field Intensity Distributions for Different Micro-Lenses.

According to the theoretical argument above, the end surface of the primary fiber should have a radius of ' ρ ', if the performance of the EFPI system is to be enhanced. It is obvious that the shape of the end surface of the fiber must be symmetric around the fiber axis, and preferably circular. Possible practical

experimental methods to fabricate this end surface were considered, and the relatively simple method of selective acid etching was attempted.

Approximately ten fiber ends were etched and inspected under a Zeiss microscope to examine the quality of the end surface. Although the end surfaces all looked curved, optical measurements performed by launching 633nm Helium-Neon laser light into the opposite end of the fiber and looking at the emerging output end pattern, which revealed inconsistent effective radii of curvatures for the end surfaces. Optimally, the end surface radius should be controlled precisely so the performance of each of the fabricated EFPI elements would be the same.

Another method attempted was forming the microlens on the EFPI with the help of a fusion splicer. This method, with constant current and fusion time settings, proved more successful than the previous method.

An experimental setup to test the microlensed EFPI is shown in Figure 2-6. A pigtailed semiconductor laser at 1300 nm was the light source. A 9/125 micron step index single-mode fiber with numerical aperture of 0.1 was used as the primary fiber. A 100/140 micron step-index multimode fiber functioned as the target fiber piece. A 2x2 tapered biconical bi-directional coupler was used to separate the input and output signals so that the sensor output could be monitored on the oscilloscope. The primary fiber was fixed onto a micropositioner to precisely control the gap between the primary and target fibers, and gap values were read from the positioner's micrometer. The microlens was formed by fusing the single-mode fiber endface using an electric-arc fusion splicer. The radius of the endface of the fiber was controlled by setting splicing time and current on the fusion splicer, and was found to be 89mm. First, the air gap between the endfaces' was set to zero and then was gradually increased by changing the positioner on which the primary fiber was mounted. The fringe contrast was obtained by measuring the voltage levels of the peak and valley fringes.. The corresponding air gap was determined by counting the number of fringes from the zero gap point on the oscilloscope screen. A similar experiment was done on a standard EFPI to compare the performances of both configurations of sensor. Figures 2-7a and 2-7b show the oscilloscope traces obtained from the micro-lensed and standard EFPI sensors. The fringe contrast versus the air gaps for the two sensors are plotted in. In Figure 2-8 it is noted that the decay rate of the micro-lensed EFPI sensor has been significantly reduced compared with the standard EFPI.

2.2.2 3-Dimensional Strain Sensor Research

No novel single-sensor design for measuring 3-dimensional strain was discovered. The only practical concept arrived at was a rosette, which is a combination of three sensors aligned at distinct angles from each other on a

Figure 2-6 - Experimental Setup for the Micro-Lensed EFPI Sensor Test.

Figure 2-7a - Oscilloscope trace of the micro-lensed EFPI output.

Figure 2-7b - Oscilloscope trace of a conventional EFPI output.

Figure 2-7 - Comparative Oscilloscope Traces for EFPI Outputs.

Figure 2-8 - The Fringe Contrast as Functions of the Air Gaps for the Micro-Lensed and Conventional EFPI Sensors.

single plane. This was not implemented into any of the simulation tests due to the limited number of detector channels and the difficulties of survivability with a single sensor.

2.2.3 500°C Anomaly in Sensor Response

A full analysis of the 500°C anomaly was found to be beyond the scope of the program. However, laboratory tests found that an initial annealing period at 500°C or higher would fix the sensor response to allow proper readings during any subsequent tests. It was, therefore, recommended to include a preliminary annealing step, either in a furnace before installation or as the initial step during the tests themselves. The latter was done for all of the simulation tests to meet the testing schedule.

2.2.4 Metal Coatings Directly on the Optical Fibers

Regular fiber coatings are usually made from organic materials which can only withstand temperatures below 300°C. Since the testing in this program requires effective operation at 850°C or higher, ceramic or metal coating materials must be used. The highest temperature coating material at the present time is gold. We, therefore, constructed gold-coated fiber-based EFPI sensors and tested the temperature characteristics of the fiber and the sensors at temperatures up to 900°C.

The gold-coated fiber used in our experiments was manufactured by Fiberguide Industries. Since the softening point of gold is about 750°C and softening temperature of fused silica, the fiber material, is about 1800°C, the highest operating temperature suggested by the manufacturer for long-term use is 750°C. Also, at high temperature >1000°C, the Germanium diffusion effect in the fiber core becomes non-negligible, and the refractive index profile of the fiber cross-section will be changed. This would result in fiber attenuation.

Because we are interested in temperatures higher than the recommended level, the gold coated fiber was tested in the laboratory at high temperature. The fiber was placed into a kiln and the temperature was raised gradually up to 900°C. The fiber remained at 900°C for two hours and then the temperature was reduced gradually to room temperature. After checking the fiber, no significant change in mechanical properties was observed.

Figure 2-9 shows the configuration of the first gold fiber EFPI sensor. The gold coating at the sensor lead was removed by dipping the fiber into aqua-regia, which was composed of three parts HCl and one part HNO₃, for about twenty minutes. To connect the gold fiber to the EFPI support box, the gold coating at the other end of the fiber was also removed using the same method.

Figure 2-9 - Configuration of the First Version of a Gold-Coated Fiber-Based EFPI.

The sensor was attached onto the surface of a ceramic filter using a high-temperature inorganic adhesive. The attachment point of the primary fiber to the ceramic filter was chosen at the edge of the gold coating, as can be seen in Figure 2-9, to prevent the fiber from being broken. The temperature of the kiln was monitored by a thermocouple, and was gradually increased. During temperature changes, the sensor outputs were monitored. The expected fringe number increase with an increase in temperature was observed, however, the fringe disappeared when the temperature reached about 400°C, and after that point no fringe variations were obtained. After this test, the primary fiber was investigated using a microscope and observed to be broken (a crack occurred across the fiber). This break might have been responsible for the disappearance of the fringe variation. This may have been induced by the roughness of the fiber surface, caused by the "corrosion" of the acid. The roughness of the fiber may have given rise to a high stress concentration which induced the crack.

To prevent fiber breakage, a second sensor was built, which is shown in Figure 2-10. The input/output fiber and the target fiber were all gold coated fiber. The outer diameter (O. D.) of the gold coated fiber was measured to be 158 μm , but the inner diameter (I. D.) of the hollow glass tube was 180 μm (this was the only tube available for this size range). The sensor was attached to the same ceramic filter and in the same way as the first sensor. The same experimental procedure was repeated. The sensor gave clear fringe outputs over a temperature range from room temperature to 900°C (this temperature change

Figure 2-10 - Configuration of the Second Version of a Gold-Coated Fiber-Based EFPI.

took about 10 hours). However, the fringe contrast in the sensor output dropped more quickly than expected. The sensor was kept at 900°C for about two hours and then the temperature was gradually reduced down to room temperature. However, during the entire cool down to room temperature the fringe contrast in the sensor output remained small (much smaller than the original value). This phenomenon could be due to the misalignment of the fibers in the tube.. To verify this judgment, the hollow tube was disturbed mechanically and the fringe contrast came back to the original value.

Several gold coated fiber-based EFPI sensors were fabricated and tested in the laboratory. The experimental results were promising. But, it was observed that fringe contrast variations existed in the sensor output that were much larger than expected for the same temperature changes. This problem was suspected to be due to the mismatch between the outer diameter (158 μm) of the gold coated fiber and the inner diameter (180 μm) of the used hollow glass tube.

Based on the experimental results obtained, we recognized that a hollow glass tube whose inner diameter matches the outer diameter of the gold coated fiber is desired to achieve good fringe contrast and repeatability. Several hollow core fibers with different inner diameters ranging from 155 to 175 μm in diameter were manufactured using the fiber drawing facilities at Va Tech. Lab

tests found 166 μm to be the optimum inner diameter for guiding the gold fiber yet allowing for freedom of movement.

A sensor built with the new hollow glass tube was attached on to the outside wall surface of a mullite candle filter segment using 905 Cotronics adhesive. Figure 2-11 shows the oscilloscope trace of the sensor output with temperature changes. In this figure, it is noted that the sensor gave a good fringe contrast for the temperature increase from 25°C to 750°C. Similar results were also obtained for a decrease in temperature. The fluctuations of the fringe amplitude in the figure is caused by laser noise. Finally it should be noted that the performance of the gold coated fiber-based EFPI sensor improved dramatically due to the use of the new matched hollow glass tube.

During the evaluation of the first in-house filter simulation test (see Section 4.2), it was discovered that the gold coating causes an expansion mismatch during thermal expansion which distorts the sensor output and causes delamination of the gold from the glass. As a result, all of the sensors used for the second and third tests had the gold stripped off in the sensor region, as in Figure 2-9.

2.2.5 Increase in the Response Rate of the Sensors

To capture transient phenomena during filter blowback cleaning procedures, which occur over a time period of about 0.1 sec., STC determined that the sensors should have at least a kilohertz response rate. Va Tech was confident that the optical fiber sensor would be capable of such a rate, although data collection had been much slower than that. They determined that the issue lied with the electronics. As a result, designs were considered for a high-speed acquisition and real-time processing system for the sensor outputs. Previously, all data collected from quadrature phase-shifted sensors were stored on a disk from a digital oscilloscope and manually processed at a later date to determine displacement, direction of displacement, and strain. An important capability for the sensor system would be to have this processing automated, preferably in real-time or quasi-real-time. The data acquisition and processing system would be most important for the outputs from quadrature phase-shifted strain and temperature sensors.

It was determined that real-time processing of the signals was beyond the scope of the contract, and efforts were directed towards effectively capturing data in two modes: a) the strain effects due to the relatively slow temperature increases and decreases and b) the strain effects due to blowback pulse cleaning cycles.

Figure 2-11 - Oscilloscope Trace of the EFPI Sensor Output with Modified Hollow Tube Sleeve.

Two important issues were addressed concerning the electrical signals obtained from the sensors. One is the indeterminacy of the initial sensor output, and the other is the experimentally observed result that the outputs of the quadrature sensor do not remain in quadrature over the full range of sensor operation. Concerning initialization, the output voltage obtained when the sensor signal processing electronics are turned on does not indicate its location on the sensor's sinusoidal voltage vs. displacement transfer function. This is due to several reasons. Exact replication of the sensors' set-point is not possible at present because the sensor is sensitive to nanometer displacements and is calibrated by hand during production. In addition, with this high sensitivity to displacement, environmental changes, especially temperature, alter the initial set-point. Another factor contributing to the issue is the fact that the sensor with electronics is a DC system. Together, these issues coupled with the variability of optical sources and attenuation in optical fibers and connectors, produce a variable output.

During experimentation, the indeterminacy problem was overcome by deliberately introducing a sufficient temporary displacement prior to a test to traverse at least one complete cycle of the transfer function. In this way the voltages corresponding to the positive and negative peaks of the transfer function were located and used as reference values, and then measurements were begun. The electronics, therefore, needed to contain variable, switchable ranges to retain sensitivity in the measurements.

The other problem has to do with the experimentally-observed result that the outputs of the quadrature sensor do not remain in quadrature over the full range of sensor operation. In fact, they could even be in-phase for some displacements. During experimentation, the quadrature phase relationship was re-established manually by changing the laser current so as to produce a change in its optical wavelength. Sufficient change would re-create quadrature for a finite range of displacement. This correction technique may not be applicable in all cases, however, and it was beyond the scope of the program to develop electronics to control the relative phase sufficiently.

The final design was a PC-based data acquisition system that would complement the data taken from the digital oscilloscope. although its function is only for data acquisition, it was designed to allow real-time data processing, as well, once the proper package was integrated into it. The PC-based interface consisted of the MetraByte DAS-20 Board along with an accessory simultaneous sample and hold box (SSH-4). Software drivers were written to operate the Direct Memory Access (DMA) interface for the DAS-20 board. Currently data is collected into a dual memory buffer arrangement. At any one time, one buffer is being used for data collection (via DMA) while the second could be used for processing. Thus far we have been able to demonstrate the simultaneous collection of data from two channels at a rate of 6500 samples/second. The

existing software program organizes the data into a form that easily allows for the implementation of required data processing algorithms.

2.2.6 Quadrature Phase-shifted Sensors

As was explained in Section 2.1, quadrature phase-shifted (QPS) sensors are important in determining the direction of strain. For the practical utilization of the strain sensors, a side-by-side dual sensor approach was chosen over a dual wavelength approach. The simplicity of a single fiber sensor, however, prompted efforts by Va Tech to develop a promising single-fiber technique, as will be explained in below in Section 2.2.7.

2.2.7 Absolute Strain Sensor

In a conventional EFPI sensor, a fringe counting technique has to be employed to recover optical phase changes (magnitude, direction) in the sensor outputs. Such a sensor can only be used to measure relative variations in the sensor gage length. However, for most industrial applications, absolute strain measurement is required. To meet this demand, Va Tech proposed a demodulation technique for the absolute phase recovery of an EFPI sensor.

The technique for demodulating the absolute sensors is as follows. If we ignore the decay of the fringe contrast in the sensor output versus the increase of the gage length, the sensor output can be approximately expressed as

$$I = I_0 \sin\left(\frac{4\pi Ln}{\lambda_s} + \phi_0\right), \quad (10)$$

where L is the distance between the two endfaces of the fibers, n is the refractive index of the material filling the gap, λ_s is the wavelength in vacuum of the ideal monochromatic source, and ϕ_0 is some initial phase. For the forthcoming analysis, the cavity is assumed to be air filled, so $n = 1$. By elongating the gap, one produces the well known sinusoidal behavior of the EFPI. The inherent drawback to this type of sensor design is that it is a relative displacement sensor. Furthermore the sensor may miss a change in direction if the change occurs at a maximum or minimum of the sinusoidal response.

Rather than set the phase difference between two distinct sensors (quadrature phase shifted EFPIs), we instead monitor the phase difference between two wavelengths in the output spectrum of a single EFPI. The absolute sensor then becomes a simple extension of the traditional EFPI. Let λ_s take on

two different values, namely λ_1 and λ_2 , where $\lambda_1 < \lambda_2$ and redefine the argument of the sinusoid to be

$$\varphi_{1,2} = \frac{4\pi L}{\lambda_{1,2}}, \quad (11)$$

then the phase difference becomes

$$\Delta\varphi = \varphi_1 - \varphi_2 = \frac{4\pi L \lambda_1 \lambda_2}{\lambda_2 - \lambda_1}. \quad (12)$$

Notice the initial phase term φ_0 has been neglected for the mathematical development. Since it is a constant effect, it can be reintroduced into the final equations to compensate for initial effects. Rewriting Equation (12) gives

$$L = \frac{\Delta\varphi \lambda_1 \lambda_2}{4\pi \Delta\lambda}, \quad (13)$$

where $\Delta\lambda$ is the difference of the two wavelengths to be scanned. By scanning the phase difference accumulated between λ_1 and λ_2 , one can apply Equation (13) to compute the corresponding gap length. The functional relationship of $\sin(\Delta\varphi)$ to gap separation and wavelength is shown graphically in Figure 2-12.

A demodulation technique and setup for an absolute EFPI fiber sensor is shown in Figure 2-13 where a white light source served as the source. An EFPI was attached to a translation stage to provide accurate displacements and the sensor output was monitored on an optical spectrum analyzer. The EFPI leads were constructed from 1300 nm circular core fiber with a cutoff wavelength of approximately 1100nm for single mode operation. A representative output scan, as well as the theoretical prediction, is provided in Figure 2-14. Notice that we were not able to equally distribute power among all of the wavelengths, which attributed to the decaying response of the experimental data. The spectral response could be calibrated with respect to an initial spectral response to offset the effects of the unequal power distribution. However the varying amplitude does not significantly affect the spectral location of the peaks. The sensor results are given in Figure 2-15. The line on the graph indicates our desired gap displacements using the translation stage. Although the translation stage has a vernier scale of 1 micron, mechanical considerations and human error limited

Figure 2-12 - Theoretical EFPI Sensor Response as a Function of Air Gap and Wavelength.

Figure 2-13 - Experimental Setup for Absolute EFPI Measurement.

Figure 2-14 - Experimental and Theoretical Response of the Absolute EFPI Sensor.

Figure 2-15 - Experimental Results of the Absolute EFPI Sensor.

the overall accuracy to a few microns. Finally, by estimating the noise associated with the amplitude of the spectral response, we computed the accuracy of the gap measurement to be 5 nm.

For this method, we are limited to static measurements because of the slow scanning speeds associated with the optical spectrum analyzer. The time to produce a scan may be reduced by more efficient coupling of the white light source (thereby allowing the spectrum analyzer to work in a fast scan mode) or by circumventing the spectrum analyzer altogether. Recent efforts related to this eliminated the spectrum analyzer and replaced it with a diffractive element and a CCD detector. With this, up to a 10kHz signal can be measured with 10 microstrain resolution. It has a 100 μm maximum gap limit with a 10mm gauge length. Va Tech estimates that such a sensor could be remotely placed 100 m away from the optoelectronics.

2.2.8 EFPI Temperature Sensors

A temperature sensor was developed based on the same EFPI technology used in the strain sensor. A number of these sensors have been built in the laboratory, but the response rate was only slightly better than 8.6 Hz.

2.3 TECHNOLOGY TRANSFER

STC successfully transferred the EFPI technology from the Va Tech laboratories to STC. The transfer included hands-on experience in the design and construction of EFPI sensors and related signal processing issues. The technology transfer was enhanced by a trip to Va Tech for their Sixth Annual Review Conference of the Fiber & Electro- Optics Research Center and the Fifth Annual Smart Structures & Materials Workshop.

During this time, we have already found a potential customer for the optical fiber strain sensor at Westinghouse. The nuclear materials testing group at STC is interested in a strain sensor to be placed in a nuclear reactor simulation chamber (hot cell) to monitor strain degradation on metal components that have had extensive exposure to pressurized water reactor conditions. As a first test, they placed small pieces of fiber into the environment to ascertain the fibers' survivability through worst-case radiation conditions. The fiber successfully survived approximately one million rads of gamma radiation. Plans are to continue the development of the sensor for this application.

3. APPLICATION TO CERAMIC BARRIER FILTERS

3.1 ISSUES

The optical fiber sensors developed in Task 2 were attached to candle and cross flow ceramic barrier filters for use in the three in-house high-temperature, high-pressure (HTHP) simulation tests. For this to take place, STC with support from Va Tech needed to resolve four main issues: 1) effective adhesives to bond the fiber sensors to the filter, 2) optimal location of sensors on the filters, 3) accurate measurement of the gauge length, and 4) methods of getting fiber leads from the electronics to the sensors.

Significant discussion was directed at the bonding issues. The proper match of adhesives with the sensors must be made to achieve sufficient bonding to the filter while maintaining an equivalent thermal expansion. From experience on the previous Smart Structures contract, Va Tech recommended Cotronics 905 as a good adhesive for the sensors. Similarly, they had found that Cotronics 989 had worked well for bonding the stainless steel tubes that protected the fiber leads.

Filter models predict where good locations for the sensors should be. The choice of sensor placement, however, was a balance between ideal sensing locations and the ability to place the sensors there. For example, many potential locations on candle and cross flow filters are located in confined, hard-to-reach sections of the filters. A good method of alignment over the location would be required in such a way as to apply the proper adhesive at the gauge length points. The actual gauge length would then need to be measured with accuracy to extract strain and stress from the data.

3.2 OPTICAL FIBER SENSOR ATTACHMENT DEVICE

STC designed a device that helped solve these last issues. It involves mounting the optical fiber sensor in a machined block of organic material, called the optical fiber sensor attachment device (OFSA). Well-defined areas on the block define the points of bonding of the sensor to the filter. The high temperature adhesive is then applied to these areas, thereby bonding together the fiber, the side of the block, and the filter surface in a controlled manner. Inside the high-temperature environment, the organic block will evaporate, leaving the sensor attached to the filter at two well-defined spots. The distance between the spots can be measured accurately before attaching the sensor to the filter by measuring the block dimensions.

The first OFSA design was a rectangular squat u-shape, as can be seen in Figure 3-1a, and was made of acrylic because it is both easy to machine and leaves no residue after being burned away at temperatures above 300°C. The design contained drilled circular channels halfway up the OFSA device to support and align the optical fibers that make up the strain sensor. The optical fiber sections were inserted into these channels and the sensor was assembled in the center space created by the squat u-shape. As can be seen from Figure 3-1a, two wells exist on either end of the OFSA device. Ceramic adhesive is injected into these wells to form two posts when the acrylic burns away, thereby defining the sensor gauge length.

Duco cement was used to bond the optical fiber sensor in the OFSA device. After the cement dried, it was noticed that the sensor gap increased, indicating that the Duco cement shrunk and pulled the sensor fiber sections apart. The increase in the sensor gap, however, did not affect the initial testing of the OFSA device. Other means of bonding was used for later OFSA versions, as is described below.

Once the OFSA device with the sensor attached was glued by Duco cement to the ceramic filter material, ceramic adhesive was poured into the two post wells. The assembly was fired for 30 minutes at 450°C to burn off the acrylic and Duco cement and to cure the ceramic adhesive. When the assembly was removed from the furnace, the acrylic and cement were gone, and the fiber sensor was suspended between the two ceramic posts. The posts were well-defined, but the fiber sensor broke soon afterwards at the points of contact with the posts. This was probably due to the brittleness of the fiber, having had its acrylic buffer coatings also burned off in the furnace.

As a result of this test, the initial OFSA design was modified in order to ruggedize the optical fiber strain gauge. The optical fiber sensor was moved closer to the filter surface to accomplish the following:

- lessen the possibility of fiber breakage,
- reduce the volume of plexiglass to be burned off,
- minimize error in the strain measurement, and
- decrease the size of the gauge length posts.

To further increase the robustness of the sensor, gold-coated fiber was used. The modified design is shown in Figure 3-1b. For this design, the optical fiber sensor was constructed first and then glued into its channel on the block. Locktite was used this time instead of Duco cement. Epoxy was then used to bond the OFSA assembly onto the ceramic filter material, and the ceramic adhesive was poured into the wells.

(a) Initial Model

(b) Second Model

Figure 3-1 - First Two Optical Fiber Sensor Attachment Device Designs.

After a similar furnace run, the OFSA device was gone, leaving the gauge length posts and the fiber sensor in between. The fiber sensor remained intact and sturdy this time and responded well to a lab bench test of the sensor response to temperature. A photograph of this model before and after the burn-off stage can be seen in Figure 3-2. As can be seen from the photograph, there was some residue left on the ceramic filter material due to the epoxy.

One final model design was made to better accommodate application in actual filters. The block size of the OFSA device was reduced from that of the previous models and two slots were machined into the block to accommodate a quadrature phase-shifted sensor, if desired. This modified design is shown in Figure 3-3. This design was tested with the same procedure as the previous versions had undergone. Both slots had sensors inserted into them, and both sensors remained intact when taken out of the furnace. Significant residue was noticed on the glass sleeve of the sensors, which was likely due to a combination of the coating put on the sleeve at manufacture and the cement used.

As is, this device would be optimally used with a filter with flat geometries like the cross flow filter. For the best use with a candle filter, the bottom surface could be made with a radius to accommodate the curvature of the filter surface.

Va Tech used OFSAs to attach sensors in hard-to-reach areas on cross flow filters. Their procedure for OFSA sensor attachment was as follows: first, the fabricated EFPI sensor was placed in the bottom groove of the OFSA, and the sensor was biased by adjusting the sensor gap. The two fiber pieces were then fixed to the OFSA by applying a small amount of superglue on the outer edges. Then the entire unit was inserted into a channel of the filter and was fixed to a side wall inside the channel with five-minute epoxy. After the epoxy set-up, Cotronics 905 adhesive was placed in the two holes of the OFSA. At the same time, the stainless steel protective tube was brought into position and bonded to the filter using Cotronics 989.

The first time the OFSAs were used with an operational sensor on a filter, it was tested to approximately 650°C. Figure 3-4 shows the sensor response before, during, and after OFSA burnoff. As can be seen from the sensor output in Figure 3-4, the signals settle to a form characteristic of normal sensor responses. A back calculation of gauge length from the sensor output yielded a length of 10.235 mm, agreeing closely with the empirical measurement of the OFSA gauge length before the test, 10.28 mm.

Figure 3-2 - The Second OFSA Model Before and After the Burn-Off Stage.

Figure 3-3 - The OFSA Design Suitable for Filter Testing.

Figure 3-4 - Sensor Response from an EFPI Sensor Attached to Filter Material by an OFSA. The arrow indicates where complete burn-off of the OFSA took place.

4. TEST AND EVALUATION

The purpose of the in-house simulation tests was to address the following two technical issues:

- 1) the ability of second-generation optical sensors in the smart filters to survive and provide useful output signals under typical filter operating conditions, and
- 2) the ability of the sensors' outputs to describe filter stressing during the typical operation conditions.

These issues were addressed to the extent of exposing the smart filters to simulated operating conditions at the existing high temperature, high pressure (HTHP) pressurized fluidized-bed combustor (PFBC) simulator test facility located at Westinghouse STC. Three tests were performed which collectively covered both cross flow and candle filters and included realistic ranges of application parameters to be expected in the field. The first two tests used candle filters and the third used a cross flow filter.

4.1 TEST FACILITIES

All three tests on the filters were conducted in the existing high-temperature, high-pressure (HTHP) pressurized fluidized bed combustor (PFBC) simulator located at Westinghouse STC. The Process and Instrumentation Diagrams (P&IDs) describing the facility are shown in Figures 4-1 and 4-2. Figure 4-1 shows the filter test vessel and the auxiliary support systems. These include two high-pressure air compressors, a natural gas compressor, a dust feed system and the associated instrumentation.

The two air compressors are capable of providing up to 1500 lb/hr of air at 200 psig. A mass flow meter and control valve are used to adjust the desired flow rate to obtain various filter face velocities (typically 2-10 ft/min). Part of the air flow is used to pressurize the dust feeder and to pneumatically transport the particulate into the filter system. The particulate is gravimetrically metered into the air stream so the inlet dust loading is accurately known.

The natural gas compressor is capable of providing up to 18 scfm of natural gas at 200 psig. A temperature controller and control valve are used to adjust the combustor temperature up to ~1800°F, at whatever ramp rate is necessary to control the temperature rise of the filters under test.

The filter test vessel is ASME coded for 350 psig at 650°F skin temperature. It is designed to accommodate up to four commercial-sized

Figure 4-1 - Schematic Diagram of the STC PFBC Simulator Filter Test Vessel Process.

(12 inch x 12 inch x 4 inch) ceramic cross flow filters or up to 19 - 1.5 meter long ceramic candle filters. A dust collection vessel is located beneath the filter test vessel to allow for long duration operation.

Figure 4-2 shows the filter test vessel again with the isokinetic sampling train, the cleaning pulse system, and the filter system instrumentation. The isokinetic sampling train is used to measure the outlet dust loading from the filter under test, in order to calculate the dust removal efficiency. The cleaning pulse system uses high-pressure nitrogen bottles to supply the pulse accumulator tank with an adjustable pressure. The cleaning pulse is timed and uses a solenoid valve to deliver the flow to the clean side of the filter, to cause a reverse flow of nitrogen through the filters. This reverse flow causes the dust cake to release and fall into the dust collection vessel.

The exhaust gas from the filter system passes through a water-cooled pipe before reaching a back-pressure control valve. The pressure is dropped across this valve, and the cooled, low-pressure exhaust is vented outside the building into the atmosphere.

The filter system is normally operated at pressures up to 150 psig with 1500-1600°F in the filter vessel. The air flow is adjusted to achieve the desired filter face velocity, and the dust feed rate is adjusted to give representative dust loadings into the filter vessel. The system is flexible enough to operate over a wide range of conditions, thus making it a good test facility to test smart structures.

4.2 FIRST SIMULATION TEST

The test preparations began on July 20 with the attachment of the optical fiber sensors onto the candle filter at the HTHP facility. Following the strategy laid out in the test plan, sensors were placed on both the clean and dirty sides of the filter. The plan was to have sensors located near the flange, in the middle, and at the end of the filter. For the attachment of the sensor on the clean side at the end, two 0.5 in. holes were drilled in the filter at the end; these holes were later plugged with ceramic adhesive after the sensor was attached. Due to time constraints and the greater-than-expected difficulty in attaching sensors on the clean side, no sensor was attached in the middle of the filter on the clean side, and there was no redundancy of sensors in any location. Also, more time was needed to solve the bonding issues associated with attaching a sensor on the flange mount, and so no sensor was placed there. Figure 4-3 shows the strategy of the sensor bonding. A hollow glass tube, the same type as was used for the optical fiber sensors, was placed at the opening of the steel tube in order to allow unrestricted movement of the fiber due to thermal expansion while

Figure 4-3 - Configuration of Optical Sensors on Instrumented Candle Filter for Test One.

simultaneously maintaining the pressure seal. Figure 4-4 shows a photograph of the completed smart filter before the test.

On Tuesday, July 21, the smart filter was loaded into the simulator and the simulator prepared for the test. In an attempt to detect strains due to the clamping of the filter into the tube sheet, Sensor 4, as labeled in Figure 4-3, was interrogated during the procedure. No signal was detected that could be attributed to strain in the axial direction of the filter.

During the filter system assembly, Sensor 2 was inadvertently broken, and so only four sensors produced data during the test.

The high temperature, high pressure test began on Wednesday, July 22. The temperature ramp was established, but it was determined that the system would not stabilize well at the lower temperatures. For this reason, no back pulse (blow back) procedures were run lower than 650°F. With the first blow back of the test, Sensor 4 failed. This left the quadrature phase-shifted sensor, Sensor 3, and Sensors 1 and 6 still operational. The simulator had difficulty below the operating level performing blow backs while still maintaining the combustor flame. As a result, the 300°F steps prescribed in the test plan were modified to try and collect useful data while still allowing the simulator to function properly. Blow backs were performed at approximately 780°F, 840°F, 1020°F, and 1090°F. When the temperature reached approximately the 850°F level, Sensors 1 and 6 degraded to the point of failure. At 1380°F, Sensor 3 failed.

The additive test continued, reaching a maximum temperature of 1617°F, before shutting down because of a possible leak. Fly ash dust had been injected into the system by that time.

This was the first test in which optical fiber sensors had been instrumented on a candle filter. Issues such as non-planar geometries, limited access locations, clean-side instrumentation, and different filter mounting and vessel environment were all addressed for the first time. Except for the breakage of Sensor 2 during installation, all of the sensors operated for at least a portion of the test and produced useful data.

Following the shutdown of the test and the removal of the smart filter from the vessel, inspection of the filter was performed, both visually from the outside and on the inside with a fiberscope. Pictures were taken of the outside, and the view from the fiberscope was recorded on video tape.

It was evident that the failures occurred in the lead fiber and not in the sensor region, which is the region bounded by the adhesive bonds that define

Figure 4-4 - Smart Candle Filter Before Test One.

the gauge length of the strain sensor. As best as one could see, the sensor regions were intact and appeared as they did before the test. The leads on Sensors 3 and 6, the sensors on the outside of the filter, experienced plastic deformation in the region between the sensor region and place where the fiber entered the steel tube. Figures 4-5 and 4-6 show before and after views of Sensors 3 and 6, respectively.

On the inside of the filter, the fiber leads of Sensors 1 and 4, which did not have any steel tubes bonded in the vicinity of the sensor region to support the fibers, broke either at the gauge length adhesive point or just before. Figure 4-7 shows an after-test view of Sensor 4.

After fiberscope video images of the region around Sensor 1 were taken, the filter was sliced up to allow closer inspection of the region. This inspection revealed that the sensor was improperly bonded to the filter. Instead of bonding the lead fiber to the filter, only the glass sleeve was, in fact, bonded to the filter, thus allowing the lead fiber to move freely, independent of any filter movement. There was a misleadingly good sensor response for this sensor at the beginning of the test, and this was likely due to the position of the fiber relative to the filter. Since it hung down the filter, gravity kept it in place inside the glass sleeve as long as no blow-back pulsing occurred. As the filter expanded thermally, the target fiber moved relative to the glass sleeve, as it should have, and the net result was an increase in separation between the two fibers, yielding a sensor response.

The processed sensor response was not what was expected. Although the sensors showed expansion while the temperature was increasing, the dilatation did not track as previous EFPI sensors had. Figures 4-8 and 4-9 show the temperature and sensor fringe response, which is a measure of expansion and contraction, inside the simulator as a function of time for Sensors 3 and 4, respectively. A comparison of the two plots do indicate, however, the relative effects of a thermal gradient through the wall of the filter. As would be expected from a thermal gradient through the filter material, thermal expansion of the outer surface of the filter is seen to lead the expansion on the inside.

The unusual sensor response lead to an analysis of the actual response of the gold-coated fiber sensor over the full temperature range of interest. The complication came with the addition of the gold coating on the silica fiber in the sensor region. To determine strain, one measures $\Delta L/L$, where L is the gauge length of the sensor. The change in gap yields ΔL . However, the change in gap is dependent on the thermal expansion of the sensor fiber, as well as the thermal expansion of the filter. Any expansion of the sensor fiber tends to reduce the gap at the same time as the expansion of the filter tends to increase the gap. What has allowed proper operation of the silica sensor is the fact that the expansion of silica is an order of magnitude less than the filter material. With

Sensor 3 Before Test

Sensor 3 After Test

Figure 4-5 - Sensor 3 Before and After Test One.

Sensor 6 Before Test

Sensor 6 After Test

Figure 4-6 - Sensor 6 Before and After Test One.

Figure 4-7 - Sensor 4 After Test One.

Figure 4-8 - Fringe Response and Temperature vs Time for Sensor 3 in Test One.

Figure 4-9 - Fringe Response and Temperature vs Time for Sensor 4 in Test One.

the introduction of the thick gold coating on the fiber in the sensing region, however, the gold will exert a force on the silica to expand more than normal. As long as the gold coating is well-bonded to the silica, the effective coefficient of thermal expansion of the sensing fiber would be altered and is unknown. It is suspected that it is somewhere inbetween pure silica and pure gold, but efforts to model the thermal expansion have not been successful due to lack of existing reference data of the materials at the high temperatures of interest. Post inspection of lead fiber subjected to the first test showed a clear delamination of the gold coating from the silica. This supports the theory that an expansion mismatch has occurred and could be the source of the problems with the data. Because of these difficulties, the subsequent two simulation tests used the original sensor design, having all of the gold stripped off the fiber in the sensing region.

4.3 SECOND SIMULATION TEST

Originally, the second test was to be a cross flow filter test. To allow enough time to instrument the filter, a cross flow filter was sent to Va Tech and instrumented with optical fiber strain sensors. However, due to a last minute scheduling change at the combustor simulator facility, the cross flow filter test was pushed back to the third test, and a candle filter was quickly instrumented with sensors for the second test.

The second simulator test, conducted December 15 and 16, 1992, followed closely to the test plan. Five fiber-optic sensors were attached to the filter: two quadrature phase-shifted strain sensors, one each on the clean side and dirty side of the filter below the flanged neck, and three temperature sensors located closer to the center of the candle. Figure 4-10 shows the placement of the sensors on the candle filter.

The temperature sensors were to have a kilohertz response rate to capture thermal transients along the inside wall of the candle filter due to blowback pulses. It was built similarly to the EFPI strain sensors with the addition of a closely-matching stainless steel tube over the hollow fiber sleeve. This tube was bonded to the primary fiber and the target fiber piece so that the thermal expansion of the stainless steel could be transduced into an optical signal. The coefficient of thermal expansion of the stainless steel is well documented, and so a temperature can be derived from its expansion.

The protective stainless steel tubing for the temperature sensors was pre-shaped into an s-like configuration in order to anchor the sensor to the wall of the filter without actually bonding it to the wall and to also prevent damage to the sensor during the blowback procedures.

Figure 4-10 - Orientation of the Fiber-Optic Sensors Attached to the Candle Filter for Test Two.

The PC-based data acquisition system was used with this test.

On the first day of the test, the temperature was brought up to 780°F and held for approximately half an hour to allow the plexiglas sensor alignment pieces to burn off. The temperature was then lowered to approximately 625°F, ramped up to 1300°F, and then lowered again to test the sensors' responses. This also allowed the initial annealing of the fiber to occur to avoid any signal anomalies.

On the second day, the test began at a temperature of approximately 335°F and was increased to an operating level of approximately 1530°F. At that temperature level, a series of three clean blow back pulse procedures were performed before any flyash dust was introduced. Flyash was then introduced and four more blow back pulse procedures were performed before ending the test.

The test was ended due to a flyash leak in the system. Post inspection revealed two cracked filters adjacent to the smart filter. It should be noted that during the series of clean pulses, a loud noise was heard within the vessel.

The data acquisition system worked well capturing both the slow thermal response and also the transient blowback response. After careful analysis of the sensor data, both from the data acquisition system and the digital oscilloscopes brought by Va Tech, it was found that the QPS strain sensors on the candle filter did not produce good quality data, although they did survive the test. This was evident from the responses they produced during the blowback pulsing. Post-test inspection revealed that the stainless steel tube associated with the clean side strain sensor was detached from the filter near the sensor region and the sensor broken.

One temperature sensor, sensor 2, consistently produced good signals throughout the test. Its response can be seen in Figure 4-11.

The temperature sensors did not exhibit the kilohertz response. The issue of appropriate materials and geometry for survivability brought about a design whose response was only slightly better than 8.6 Hz. Although Virginia Tech researchers feel that better materials can be found, Westinghouse feels it is doubtful that 1 kHz can be achieved without a significant change in the sensor design.

Figure 4-11 - Temperature Response of Optical Fiber Temperature Sensor 2 Compared to a Passage Temperature Thermocouple During Test Two.

4.4 THIRD SIMULATION TEST

STC determined that the best strategy for the cross flow smart filter test would be to concentrate the sensing on one area of the filter and to take advantage of the OFSA devices by attaching sensors inside the dirty and clean channels. A corner area on one of the faces of the filter was picked, and a combination of quadrature phase-shifted and single sensors were attached in close proximity to each other. In the final configuration, two single strain sensors were placed in channels on the clean side, and two more were placed opposite them in dirty side channels. OFSA devices were used to attach these sensors to the filter. Three other sensors were manually attached to one of the faces of the filter on the dirty side. All three of these were quadrature phase-shifted (QPS) sensors. Figure 4-12 shows the locations of the sensors on the first instrumented cross flow filter.

Once the third test was announced, Va Tech prepared the filter by heating it in a university kiln to burn off the optical fiber sensor attachment (OFSA) devices. However, in doing so, a large crack formed in the filter. The temperature profile was reasonable for heating a cross flow filter, according to Westinghouse engineers, and the reason for the failure is still unknown at this time.

At the same time, Va Tech researchers noticed that the sensors did not bond well to the filter. It was suspected that the ceramic adhesive was not placed properly in the OFSA or that the filter surface was not cleaned properly beforehand. Another suggestion was that the expansion of the OFSA before it evaporated was of enough force to weaken the Cotronics bonds. Although that was not detected in the STC laboratory tests of the OFSA, Va Tech took the precaution of notching a strain relief in the remaining OFSAs at the bottom center perpendicular to the groove direction.

Another filter was then located and sent to Va Tech for another process of instrumentation. Va Tech made efforts to attach the sensors more robustly this time. The intention was to more securely attach the sensors and more securely anchor the stainless steel tubing to the filter with the ceramic adhesive. The configuration of the sensors was the same as the first filter.

The third simulator test was conducted on January 21 and 22, 1993. This test was run in combination with another cross flow filter test at the hot gas cleaning facility. In all, four cross flow filters were mounted on the tube sheet.

On the first day of testing during the initial pressurization of 50 psi at room temperature, pressure leaks were detected in the stainless steel tubes

Figure 4-12 - Placement of Sensors on the Cross Flow Filter for Test Three.

supporting sensors 2 and 5. The vessel was depressurized, and these leaks were plugged using 5-minute epoxy. Also, from the onset, the only sensors to produce signals were sensor 1 and one leg of sensor 4.

The combustor was then pressurized again to approximately 100 psi, heated to approximately 850°F, cooled to under 600°F, reheated to approximately 850°F, and then cooled once more. This procedure was done to verify that the operating sensors tracked the thermal dilatation of the filter. As an example, the change in sensor signal from sensor 1 during the first transition from heating to cooling can be seen in Figure 4-13. It should be noted that the leading signal trace of the QPS sensor becomes the lagging trace approximately half way through the figure. It was at that point that the temperature began to decrease, causing the filter to contract.

Figure 4-14 shows the sensor 1 response throughout the testing of the first day. During the first cool-down period of the test, one of the legs of sensor 1 appeared to get stuck, producing no noticeable signal in a few degree range of 620°F. The sensor leg then became active again as the temperature dropped beyond this range. On the second temperature increase, the same phenomenon occurred in a few degree range of 630°F. This occurred a third time in the same region when the combustor was cooled for the second time. These points are indicated in Figure 4-14 by the numbers 1, 2, and 3.

On the second day of testing, the combustor was heated to approximately 1540°F from a residual temperature of 305°F. Figures 4-15 and 4-16 show the responses of sensors 1 and 4, respectively. Note in Figure 4-15 that sensor 1 failed at a temperature of approximately 1350°F. Also, the figures indicate that both sensors experienced a large rate of sensor gap separation, sensor 4 beginning at approximately 900°F and sensor 1 at approximately 1100°F. Since the sensors were placed across clean-side filter laminate seams, this data seems to indicate that a delamination occurred, starting from the bottom of the filter. Post-test inspection of the filter did not show any delamination in that area, however. Once the combustor simulator reached a level of 1540°F, a series of blowback pulses were performed to monitor the response of the remaining leg of sensor 4. From this point, the filter continued to be tested as part of the other concurrent test, but the sensors were not monitored.

At post-test inspection, we found that all of the cross flow filters in the test cracked. The smart filter had a vertical crack in the middle of the filter, a significant distance away from the sensors. The stainless steel tubes were detached from their ceramic adhesive bonds on the filter, except for sensors 2 and 4. Then, as the tube sheet with the filters was lifted out of the combustor simulator, the stainless steel tube associated with sensor 2 slid freely out its ceramic adhesive bond.

Figure 4-13 - Raw Output form Sensor 1 During Thermal Cycling of Test Three.
Note the change in relative phase of the signals, which corresponds to a reversal in thermal gradient.

Figure 4-14 - Response of Sensor 1 During the First Day of Test Three. The numbers 1, 2, and 3 correspond to points where one leg of the QPS sensor temporarily stopped.

Figure 4-15 - Response of Sensor 1 During the Second Day of Testing of Test Three.

Figure 4-16 - Response of Sensor 4 During the Second Day of Testing of Test Three.

In preparing to inspect sensor 1 in an attempt to determine the cause of its "sticking" behavior during the test, the fiber sensor pieces broke into smaller pieces and were contaminated with the flyash that was on the filter. This prevented any proper inspection of the sensor.

5. SENSOR RUGGEDIZATION

Following the three in-house tests, it was concluded that further work was needed to make the sensors more rugged, both in the sense of the sensors surviving and operating throughout the tests and also keeping the sensors bonded to the filter substrates. The additional two tasks were added to the Base Program to address these issues. The first task addressed the actual ruggedization, and the second performed an in-house test using the results of the ruggedization effort.

5.1 ALTERNATIVE SENSOR LEAD FIBER COATINGS

The objective of this subtask was to examine methods of improving silica optical fiber coatings which could protect the optical fiber sensors and sensor leads from breakage in the harsh environments encountered in coal fired power generation facilities. The gold coatings used in the in-house simulation tests, while being the best high-temperature coatings available on the market, demonstrated expansion mismatches with the silica fiber. This seemed to create complications in the actual thermal expansion of the sensor, and did cause delamination of the coating from the fiber. Once a coating has detached itself from an optical fiber, the fiber becomes susceptible to mechanical failures. The sensor's survivability is therefore directly related to the durability of the fiber's coating.

Va Tech utilized rf magnetron and dc magnetron sputter-coating systems to investigate special metal coatings for optical fibers subjected to high-temperature harsh environments. The rf magnetron is a stand-alone sputter-coating system which can deposit coatings on optical fibers and other prepared substrates up to 18 inches in length. The dc magnetron sputter-coating system is a newly installed system placed in-line onto their optical fiber draw tower that can produce coating during fiber manufacture and is, therefore, not limited in fiber length. This dc magnetron sputter-coating system is a state-of-the-art system, and part of its development was accomplished through this contract's efforts. Therefore, a description of the system will be given.

5.1.1 DC Magnetron Sputtering System

The on-line optical fiber dc magnetron sputter-coating system, recently developed by Hughes Aircraft Company, now installed on the Va Tech 8-meter optical fiber draw tower, differs from other dc cylindrical magnetron sputter systems that have deposited metal coatings on optical fibers. Earlier designs have deposited coatings on previously drawn fibers. The on-line dc magnetron

sputter-coater at Va Tech is unique in that it allows for a uniform metalized coating to be applied as the optical fiber is manufactured.

The sputtering system as delivered consisted of a vacuum chamber containing 13 stacked sets of sputter targets with 7 sets in a lower bank electrically isolated from the 6 sets in the upper bank. Each set contains 3 sputter targets arranged in an equilateral triangle to provide symmetrical coverage of the passing fiber. Each target contains a NdFeB magnet with a field strength of 10500 Gauss permitting higher pressure operation of the system. The optical fiber is inserted into Argon screened, glass capillaries at top and bottom of the chamber as the fiber is pulled from the furnace. The capillary plates are then fixed to the chamber and a vacuum of 40 to 80 mTorr is developed while a fiber-draw, from a 10 mm preform, continues with parameters typical of those shown in Table 5-1.

Draw furnace (amps/volts)	Feed rate (mm/min.)	Draw rate (cm/sec)	Fiber OD (μm)
450/25	0.06	0.25	440

Table 5-1. Draw Parameters

As the diameter stabilizes the chamber pressure becomes low enough to initiate a 15 to 30 minute glow discharge for removing metal oxides that have formed on the surface of the targets after exposure to air. When the plasma stabilizes at a current of 3 amps, a sputtered coating of 1 to 3 μm having the same characteristics as the target material is deposited on the optical fiber as it passes downward through the chamber. A uniform coating is obtained despite the preferential ejection of nickel from each target. This is because the enrichment of the other components are then preferentially sputtered after the nickel is depleted. Originally the system had differential cooling between the upper and lower target banks, but the recent improvements accomplished during this project greatly ameliorated the target cooling/electrical problems, thereby helping to maintain a steady state condition preventing uneven diffusion or evaporation of the target elements.

A photograph of the dc magnetron sputter system mounted on the Va Tech draw tower is shown in Figure 5-1. Argon is used to isolate the vacuum chamber preventing contamination and to supply the inert gas ions necessary to generate the plasma. Choosing Argon as the inert gas was most cost effective even though the sputtering yield could possibly be doubled if gases such as krypton or xenon were used. The entry and exit capillary tubes through which the fiber passes were designed with proper tolerance and length to insure integrity of the vacuum system as the fiber passes through the chamber. The capillaries are sufficient in inner diameter (outer diameter of the

(a)

(b)

Figure 5-1 - (a) Va Tech Draw Tower and (b) Detail of On-Line Sputtering System.

fiber plus 18 to 45 μm) at a length of 75 mm to allow the vacuum within the system to be maintained while the optical fiber is being manufactured.

During this project there have been several major changes made to the operational components of the dc magnetron sputtering system. The areas redesigned brought about improvements over the original Hughes design and contributed to a more efficient operation of the system. Changes were made to the target cooling system, power distribution system, and the vacuum system.

The original cooling configuration was designed to provide adequate coolant to the targets in an orientation where the feed-through collar was mounted at the top of the vacuum chamber. In this configuration, the incoming, and exiting coolant lines connected to the feed-through collar. This allowed for the coolant to flow from the top down through each column of targets by the forces of gravity and pressure, allowing the air to escape at the top through the exiting line. However, the placement of the chamber on the Va Tech tower, required that the feed through collar be on the bottom of the vacuum chamber to allow for lowering the target assembly for maintenance. This orientation created air pockets at the upper end of the target assembly preventing cooling at these areas. This was evident by the dark discoloration seen at the upper target fixtures and the presence of higher temperatures noted at the top after operation of the system. This problem was corrected by tapping into the top of each column of targets and feeding the coolant line to the top through to the bottom where it exited. This change in the coolant configuration initially solved the problems stemming from the differential target temperatures, however, other better improvements have been made as a result of the redesign to solve problems associated with the electrical feed.

With the differential cooling problems solved it was thought that the changes might correct some of the problems seen in the electrical system, namely energy robbing target back discharge and arcing. In an attempt to minimize the plasma discharge at the rear of the targets, the coolant/conductor lines were shielded with a Teflon sleeve. The Teflon shields decreased the problem of the back discharge, but it did not solve the problem of arcing at the rear of the target heart and the anode base structure. Even after many thorough cleanings and extended vacuum pull-downs to allow for removal of possible arc causing contaminants, a system failure would occur due to the arcing between one of the 39 cathode-anode fixtures. Replacement of the cooling lines and fixtures, originally brass and copper, with electrical grade Teflon fittings became necessary. Also, the rear glass-filled Teflon insulators, which originally covered only the back circumference of the cathode, were replaced with ones that cover the entire back area of the cathode. The removal of the brass coolant fittings, which acted as conductor tracks for the targets, eliminated massive conductor areas at the rear of the targets thus defeating potential arcing or plasma discharge.

To originally balance the current to the individual upper and lower target assemblies, the external line resistance was tied to a dielectric plate with the high voltage resistors mounted in a series/parallel combination giving the required resistance values. The value of the line resistance varied depending on the pressure, argon concentration, target material, and attitude of the target fixture. This required that the resistor network be removed and the combination of resistors be restructured to correct for the changed line resistance. To eliminate this handicap a special resistor structure was built that allows for resistance compensation by means of rheostats and that no longer requires removal of the network from the tower.

Another area of great concern is the entrance and exiting capillaries of the vacuum chamber. The capillaries are required to maintain operating pressures inside the vacuum chamber while allowing the fiber substrate to pass through with minimal resistance. The original capillaries supplied by Hughes were tapered in diameter, so that only a small portion of the capillary came in contact with the fiber, and they created large losses in vacuum pressure. This was corrected by using capillaries that held a consistent inner diameter throughout the length of the capillary, as mentioned above, but the problems of fiber abrasion and static buildup due to the glass on glass contact are still being addressed.

5.1.2 High Temperature Coatings

The coating process and the characteristics of the buffer coating material are major factors that determine the reliability of silica optical fibers in high temperature environments. The dip process, a standard procedure used successfully to apply gold and other metal coatings such as indium, tin and aluminum, cannot be used for higher temperature metals needed for the high temperature fossil energy applications. With the dip process, the metals are applied at their melting temperatures and then cooled rapidly, inducing microbend attenuation and/or non uniformity of the coating in cross section. In contrast, vacuum deposition processes can deposit a wide range of high melting point metals at ambient temperatures to produce high strength, hermetically sealed optical fiber for high temperature applications. The rf and dc magnetron sputter coaters were used to deposit these types of coatings on the fiber.

5.1.3 Types of Coatings

Two coating materials, INCONEL 625 and Haynes 214, were selected after consideration of their melting points and their ability to adhere to silica. The characteristics of the applied sputtered coatings were compared with the target material using scanning electron microscopy (SEM), Auger electron

microscopy, and energy dispersive X-ray spectroscopy (EDAX). After these comparisons, the various coated fibers were examined for stress corrosion from thermomechanical forces encountered during temperature cycling.

INCONEL 625 is a nickel-chromium-molybdenum alloy with niobium added to provide strength; Table 5-2 displays its chemical composition. The alloy is highly corrosion resistant and has a melting temperature starting at 2350° F. This high strength alloy has applications in chemical, aerospace and nuclear engineering. The corrosion resistance and high temperature capabilities were among the prime factors for including INCONEL 625 as one of the initial test materials.

Element	%		Element	%
Ni	58.4 %		Mn	0.5 %
Cr	21.0 %		Al	0.40 %
Mo	9.0 %		Ti	0.40 %
Fe	5.0 %		C	0.10 %
Nb	3.65 %		S	0.02 %

Table 5-2. Chemical Composition of INCONEL Alloy 625

Haynes alloy number 214 is a nickel-base alloy having excellent oxidation resistance at temperatures of 2200°F. This resistance to oxidation stems from a thin aluminum oxide layer, enhanced by a small amount of yttrium, which forms on the surface of the material. This film protects the alloy from oxidation during prolonged periods at high temperature. The film also serves to increase the resistance of the alloy to carburization, an important factor in the coal gas purification environment where there would be a high presence of carbon in the filtering chamber. The chemical composition of Haynes 214 is presented in Table 5-3. The chemical composition of the alloys was used as the baseline for the composition analysis performed on the sputtered coatings.

Element	%		Element	%
Ni	75.0%		Si	0.2 %
Cr	16.0 %		Zr	0.1 %
Al	4.5 %		C	0.05 %
Fe	3.0 %		B	0.01 %
Mn	0.5 %		Y	0.01 %

Table 5-3. Chemical Composition of Haynes 214 Alloy

5.1.4 Thermomechanical Test Results

Both the INCONEL 625 and the Haynes 214 alloys were rf and dc sputtered onto silica optical fiber and the resulting coatings were analyzed. The SEM photographs, shown in Figures 5-2 and 5-3, show the deposited films at a magnification of 2020x. Note that in comparison, the coatings produced by the dc magnetron appear to be much more uniform than those by the rf magnetron. The surface of each coating was scanned, using EDAX, to determine its chemical composition. Table 5-4 was developed from the results of the EDAX measured composition of the coatings for both the INCONEL 625 and Haynes 214 alloy coatings. A comparison of the manufacturer's chemical composition of the alloys and the sputtered films in Table 5-4 demonstrate that the dc sputtered films are very similar to the target material, indicating that a true alloy had been deposited. The rf coatings, however, were not as close to the target film composition. In particular, a large preferential deposition of iron was found in both of the coatings.

Element	INCONEL 625 target	DC INCONEL 625 coating	RF INCONEL 625 coating	Haynes 214 target	DC Haynes 214 coating	RF Haynes 214 coating
Ni	58.4 %	62.7 %	42.9%	75.0 %	75.4 %	51.0%
Cr	21.0 %	26.5 %	21.3%	16.0 %	17.4 %	15.9%
Fe	5.0 %	5.6 %	33.3%	4.5 %	3.6 %	30.6%
Mo	9.0 %	5.2 %	2.2%	—	—	—
Al	—	—	—	4.5 %	3.7 %	2.3%

Table 5-4. Chemical Composition of Sputtered Alloy Coatings

The cylindrical uniformity of the dc magnetron deposited film was investigated using a laser micrometer, with a resolution of 0.01 μm . The coated samples were first evaluated by measuring the fiber diameter at various points along the fiber length. In each case the film uniformity varied < 0.1 μm .

After the coated fibers were examined using the SEM and EDAX techniques, mechanical testing of the samples was performed both before and after thermal cycling. Selected samples of the fibers were sent to STC, where they were first tested by lightly scoring the coating while being viewed under a microscope. The tip of an exacto knife was run axially along each fiber, and the fiber was observed for any damage. The dc-sputtered Inconel coating came off easily, while the rf-sputtered samples all remained intact.

The fiber samples were then placed in a single-zone tube furnace, and its temperature was ramped up to 1600°F with a rate of 60°F/min. The fibers were

Figure 5-2 - SEM of RF Sputtered Coatings. (a) INCONEL 625 and (b) Haynes 214.

5-8 67

(b)

(a)

(a)

(b)

Figure 5-3 - SEM of DC Sputtered Coatings. (a) INCONEL 625 and (b) Haynes 214.

then kept at 1600°F for 20 minutes, after which the heater was turned off and the furnace cooled to room temperature. Inspection of the fibers revealed signs of flaking and sections of the coatings missing. When tape was applied to fibers and then taken off, all of the coatings lifted off easily.

Va Tech followed up with similar tests. An adhesion tape test was conducted before thermal cycling on the coatings with no loss of coating to the tape. Then, samples of the Haynes 214 and INCONEL 625 alloy coated fibers were temperature cycled using an Isotemp Programmable Ashing Furnace. The temperature was elevated to 1652°F, held for 20 minutes and then returned to room temperature using the schedule shown in Table 5-5. Each of the fibers was subjected to a tape adhesion test after being removed from the furnace and, if possible, microscopically examined before and after each temperature test.

Step	1	2	3	4	5	6
Ramp (°C/min.)	7.2	7.2	7.2	7.2	7.2	7.2
Temp. (°C)	572	1112	1652	572	212	77
Soak (min.)	10	10	20	10	10	NA

Table 5-5. Ramping Profile for Thermomechanical Testing of Coated Fibers

Post inspection of the fibers showed that the metal coatings were scored with cracks dividing the coating into individual sections. Figure 5-4 shows before and after temperature cycling photos for two dc magnetron sputtered INCONEL coatings. The results for the Haynes 214 coating are very similar to those for the INCONEL 625.

The coated fibers were subjected to the same adhesion test as before the temperature cycling but loss of adhesion was discovered. Several factors may have contributed to the coating's crazing and loss of adhesion. The adhesion loss may have resulted from the formation of an expanding intermediate layer forming between the fiber and coating due to contamination of the substrate. The observed crazing of the coatings contributed to the poor adhesion and may be associated with the formation of a metal oxide which rendered the coatings more brittle. A similar explanation is that the increase in fragility may be the effect of the formation of the gamma prime Ni₃Al precipitates forming at temperatures between 1100 to 1750°F. While the formation of this precipitate strengthens the alloy, it also causes it to shrink and produces a loss of ductility. A loss of ductility does occur with the probable loss of carbon occurring during

(a)

(b)

(c)

(d)

Figure 5-4 - Surface of INCONEL 625 Coated Optical Fibers: Thermal Cycling Test.
Sample One: (a) before and (b) after; Sample Two: (c) before and (d) after.

the sputtering process. Also the small grain size implies that the sputtered film varies from the original lattice relating to an unknown changed thermal coefficient of expansion.

5.1.5 Alternative Coatings

Because of the difficulties with the steel alloy coatings, other coating materials have been studied for their application to high-temperature optical fiber sensors. Palladium and titanium have been identified for their superior adhesion to silica fibers at high temperatures. Va Tech has deposited palladium on silica fibers using their rf magnetron. Samples sent to STC were evaluated in a similar way that the steel alloys were tested, as described in Section 5.1.4 above. Using an exacto knife, it required a few passes with the knife to lift small amounts of the coating off the fiber, and none of the cracking and spalling experienced with the steel alloys was evident. After the thermal cycle, small areas of the coating lifted off, totalling approximately 10% of the entire length of the fiber. This was also a significant improvement over the previous efforts. When tape was applied to and removed from the fibers, no noticable amount of the coating was stuck on the tape.

These metals, however, are expected to be fairly susceptible to corrosion and/or hydrogen embrittlement. It is possible that the use of thin layers ($< 1\mu\text{m}$ thick) of these metals could act as a buffer layer which will promote adhesion between an optical fiber and a second metal.

Another effort performed under this investigation was the revisitation of the gold-coated optical fiber. Research reviews and conversations with materials experts at Va Tech indicated that annealing the gold coating may provide the strength and adhesion necessary for use in this program. The annealing process relieves quenching stress in the coating, normally caused by the rapid cooling during deposition, by allowing both the fiber and the coating to cool at the same rate.

Va Tech annealed several samples of gold-coated fiber and then made side-by-side comparison tests of bending and coating adhesion. Both sets of fibers showed good survivability to bending, down to a diameter of 5.5 mm, and the annealed fiber demonstrated more resistance to cracking and flaking. STC found similar results before and after a thermal cycle test. It was noticed that the gold coatings were thicker than those used previously in the in-house simulation tests, and that could have added to its durability.

Another possibility for durable, high-temperature coatings is the use of high-temperature oxides. Planned modifications to the preexisting dc planar magnetron sputtering system would allow researches at Va Tech to investigate a number of high temperature oxide coatings. They have identified several high

temperature oxide materials which are noted for their resistance to corrosion and which will more closely match the CTE of fused quartz. Oxides currently under consideration include 1) magnesium oxide (MgO), 2) titanium oxide (TiO₂), 3) zinc oxide (ZnO), 4) tin oxide (SnO₂), 5) aluminum oxide (Al₂O₃). Table 5-6 compares the physical properties of these oxides with those of silica.

Techniques for sputtering thin films of ZnO are well documented. ZnO, a piezoelectric material, has a CTE ranging between $2.1 \times 10^{-6}/^{\circ}\text{C}$ and $4 \times 10^{-6}/^{\circ}\text{C}$ depending on the crystal orientation. Polycrystalline thin films of ZnO on glass can be formed at a rate up to 0.5 mm/hr in conventional dc magnetron systems. Others have investigated the environmental stability of Al₂O₃ and TiO₂ deposited through reactive sputtering and in addition to exhibiting good thermal characteristics, these oxides have the applications for optical filters and antireflective coatings.

Oxide	Melting Point ($^{\circ}\text{C}$)	CTE ($\times 10^{-6}/^{\circ}\text{C}$)	Temp.range ($^{\circ}\text{C}$)
Silica	1683	0.55	--
MgO	2800	14.0	20-1400
TiO ₂	1855	8.19	0-500
ZnO	1975	2.1-4.0	--
SnO ₂	1625	4.0	0-800
Al ₂ O ₃	2046	8.0	20-1400

Table 5-6. Physical Properties of Oxides

5.2 HIGH TEMPERATURE ADHESIVES

Another factor attributing to the failure of the optical fiber sensors in the in-house simulation tests was the debonding of the stainless steel tubing from the filter material. This subtask was to determine the optimum adhesive material(s) to maintain the bonding throughout the tests. Because the Haynes 214 and INCONEL 625 coatings were under investigation for the optical fibers, the properties of these metals were taken into consideration, as well.

When choosing an adhesive for use in any temperature-variant environment, the coefficient of thermal expansion (CTE) must be considered. The CTE of silica is $0.5 \times 10^{-6} \text{ cm/cm}/^{\circ}\text{C}$, the CTE of the mullite filter material is $5.4 \times 10^{-6} \text{ cm/cm}/^{\circ}\text{C}$, and the steels have CTEs varying from approximately $13.0 \times 10^{-6} \text{ cm/cm}/^{\circ}\text{C}$ to approximately $17.0 \times 10^{-6} \text{ cm/cm}/^{\circ}\text{C}$. For the best

bonding, an adhesive with a CTE that falls somewhere between the largest and smallest CTE value should be chosen.

Va Tech conducted tests on three candidate ceramic adhesives: Aremco Products Inc. 670 adhesive, Cotronics Corporation 989 adhesive, and Master Bond #18 adhesive. They were chosen because they are alumina-based, similar to the alumina-based mullite filter material. The three adhesives were evaluated for bonding gold-coated optical fibers and 0.183 cm outer-diameter stainless steel tubes to mullite filter samples. The gold-coated fibers were used instead of the metal alloy-coated fibers due to the difficulties in producing a satisfactory metal alloy coating and because of the recent success in pre-annealing the gold-coated fibers.

The actual testing was performed in two phases: 1) tests with the optical fibers and then 2) tests with the stainless steel tubes. In each case, after cleaning the filter samples and the fibers or tubes, and arranging the fibers or tubes on the filter samples, the adhesives were applied and cured. In the case of the Aremco 670 adhesive, as suggested by an engineer at Aremco Products Inc. and by the literature associated with the adhesive, a thinner was applied to the filter surface before the adhesive to dissuade the porous mullite filter from absorbing the binder of the adhesive. Figure 5-5 shows a filter sample using the Aremco 670 adhesive to bond gold-coated fibers to the mullite filter.

Six gold-coated optical fibers were bonded with each adhesive on the filter samples. Each filter sample underwent eight temperature cycles, each cycle exposing the samples to a three-hour temperature ramp up to 1000°C and then allowing the samples cool to room temperature. After each cycle, the samples were visually examined for cracks and other imperfections and subjected to strength tests. During the strength tests, the fibers were manually pulled along their axes, and the surface of the adhesives was abraded in a direction perpendicular to the length of the fibers.

The gold-coated optical fibers were well-bonded to the filter samples by both the Cotronics and Aremco adhesives over the full eight temperature cycles with no apparent cracking or loss of adhesion by the adhesives. The Master Bond adhesive, however, failed to hold its bond from early in the testing. In summary, one fiber was loose enough to slide out of the adhesive directly after the cure, two of the fibers fell off of the filter sample after the first temperature cycle, two more fell off after the second temperature cycle, and the last fiber fell off after the fourth temperature cycle. Because of this, the Master Bond adhesive was not used in the stainless steel tubing test.

The same procedure was followed for the stainless steel tubing, except that only five temperature cycles were performed. After the last two

temperature cycles, it was noticed that the Aremco 670 adhesive loosened around three of the tubes, with one tube able to slide back and forth within the adhesive along its entire length. Va Tech noticed no deterioration in the bond with the Cotronics 989 adhesive. Cracking was noticed in both adhesives, it

Figure 5-5 - Gold-Coated Optical Fiber Bonded by Aremco 670 Adhesive to Mullite Candle Filter Section.

did not appear to affect their bonding capability. It was, therefore, Va Tech's conclusion that the Cotronics 989 adhesive was the adhesive of choice and was sufficient for this application.

STC was not satisfied by the Va Tech results, however. It was, in fact, Cotronics 989 adhesive that was used in the in-house simulation tests at STC and which failed to hold the stainless steel in those tests. It was felt that a better strategy would be to layer a series of ceramic adhesives, the bottom layer having a CTE close to that of the filter and the top layer being more suited to match the characteristics of the steel. Aremco had a wide assortment of adhesives available, and after discussions with STC scientists and Aremco engineers, a suitable combination of adhesives was determined. The bottom layer was a thin layer of Aremco 503, followed by thin layer of Aremco 517. On top of this, the bulk layer of Aremco 517 would bond the stainless steel tube to the rest.

6. IN HOUSE TEST WITH SENSOR RUGGEDIZATION

An in-house test was planned to study the performance of the sensors after the sensor improvements were completed. It was felt that another test in the HTHP facility was not cost-effective at this stage, and so the test was planned for the High Temperature Flow-Through Alkali/Ceramic Test Facility. This facility is a four-inch diameter, four - to - six feet long flow-through tube reactor that has provisions for introducing variable alkali concentrations into the reaction chamber through the use of a water-cooled salt water injection probe. The salt solution concentration and flow rate are used to control the vapor phase alkali concentrations that the ceramic filter sample experiences. It also has provisions for pulse cooling, simulating the effects of blowback pulsing on the filters samples. The flow-through capability is designed to simulate ceramic barrier filter operating temperatures, up to 1800°F. Figure 6-1 shows a schematic drawing of the facility.

6.1 SENSOR CONFIGURATION

Gold-coated optical fiber was used for this test since the metal alloy coatings had not been developed sufficiently and the new improvements in the gold coating's performance were discovered. Two sets of quadrature phase-shifted fiber sensors were built, one set bonded to either side of a ceramic disc made of filter material. The sensor sets were placed directly opposite each other in order to detect differential expansions of the sample. OFSAs, similar to those described in Section 3.2, were used to attach the sensors to the ceramic filter disc.

Ceramic discs are mounted perpendicular to the axis of the furnace, as can be seen in Figure 6-1. The discs are 2.75 inches in diameter, but only approximately 2.25 inches are available because of the ring-shaped mounting assembly. This allowed for a one-inch gauge length for the sensors plus just enough room for the sensor leads to be redirected out of the furnace through an available port tube. The tight 90° bend of the fibers necessitated the design of a special stainless steel support to prevent fiber breakage during the tests. One of the supports is shown in Figure 6-2 mounted to the ceramic disc. The design included a flanged section to help prevent sliding of the tube, even if the tube were to detach itself from the ceramic adhesive. It also incorporated a steel sleeve on the outer end of the support that mated with the port tube which protects the fibers until they exited the furnace. This created continuous support of the fiber leads from the sensor area to the outside. The initial thin layers of ceramic adhesive in the sensor area can also be seen in the figure.

The same system of detection and storage of the sensor signals as the previous in-house simulation tests was constructed. Sensor support systems

Figure 6-1 - Schematic of the Westinghouse STC High Temperature Flow-Through Alkali/Ceramic Test Facility.

Figure 6-2 - Stainless Steel Support Tube Bonded to Ceramic Filter Disc Prior to Sensor Attachment.

provided the optical power and also detected and amplified the sensor signals. These signals were then directed to a digital storage oscilloscope.

6.2 SENSOR INSTALLATION

The fiber leads from the sensors facing the down-stream side of the furnace were first fed out of the mounting chamber before the chamber was inserted into the furnace. Then, while the chamber was being inserted into the furnace, the other set of fiber leads, coming from the sensors facing the up-stream side of the furnace were fed through the available tube leading out of that side of the furnace. Near the end of the installation, however, in the process of adjusting the port tubes that mate with the fiber lead supports, a crack was heard, and we detected no signals on the down-stream side sensors. Post-test inspection revealed the support piece detached from the filter with pits in the disc where filter material came off with the tube and adhesive.

6.3 SENSOR TEST

It was decided to attempt a test with the remaining QPS sensors to test survivability. A temperature cycle was begun to verify the operation of all the systems. Soon after the temperature cycle began, the remaining sensor signals disappeared except for a small, slowly varying signal. Not sure if the sensors had, in fact, failed, we continued the temperature ramp to approximately 400°C. With no appreciable change in the sensor signal, we determined that the sensors did fail. Post inspection revealed that the other support assembly had come off the disc, this time leaving much of the adhesive on the disc.

6.4 TEST ANALYSIS

It is apparent that the adhesion problem is not solved yet. More ceramic adhesive could have been used to attach the support tube to the filter disc in the hope that more ceramic mass might retain the bond. One issue which this test might have introduced which was absent in the other tests was the possibility of a significant torque applied to the bond from the attached port tube. Since there was at least three feet of tubing from the sensor position to the end of the furnace, its weight might have attributed to at least the second detachment. It is likely, if this were the case, that the initial movement of the tubes during installation could also have applied sufficient torque to tear off the first support tube from the filter disc.

7. CONCLUSION

It is evident from all of the in-house simulation tests that the attachment and protection of the sensors on the filters is still an issue. Filter dilatation, both expansion and contraction, has been monitored well while the sensors were intact, but attempts to retrieve true stress/strain information of the material has not yet been achieved. This has been due to the failure of critically-placed sensors once the tests began. Almost all of the failures were caused by problems in securing the stainless steel protection sleeves to the filters or otherwise anchoring the fibers before the sensing region.

The smart filters are not yet ready for field testing. True strain or stress measurement has not yet been demonstrated. An engineering effort is required to design a more robust sensor system. Such a task is predicted to have a high probability of success. Also, it would be wise to consider new designs for the temperature sensor in order to increase its response rate. Richard Tressler, an STC consultant from Penn State University, has suggested that more information might be gained with temperature sensors than with strain sensors.

Much has been learned about these optical fiber sensors during this contract. Improvements have been made to the sensors with no indications that they themselves could not be made to operate in this application, if it were not for the adhesion problems. Also, research into the manufacture of high temperature silica optical fibers has made great strides with the installation of the dc magnetron in-line sputtering system. This will have an impact beyond the ceramic filter sensor efforts for fossil fuel production. Vacuum-deposited metal coatings on optical fibers could have numerous applications. They include: high temperature-high reliably cabling used in aircraft/spacecraft data transmission, fire detection systems, under sea cables, tethered vehicles and electro-optical coax, fiber optic sensors found in engine monitoring like the NASA high T hypersonic sensor development, manufacturing process control, medical probes and smart skins, encompassing air and spacecraft as well as civil and energy related structures. Besides the capability for high temperature operation, these applications would benefit from the advanced fiber coatings by having chemical/corrosion compatibility or resistance, solderability, and conductivity. Continued development of the on-line process will certainly advance these applications and create new ones. Exciting future generations of special coatings could incorporate shape memory alloys, magnetostrictive, electrostrictive, and electrically restrictive materials.