

A Development of On-Line Temperature Measurement Instrumentation for Gasification  
Process Control

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## Abstract

This progress report covers continuing work to develop a temperature probe for a coal gasifier. A workable probe design requires finding answers to crucial questions involving the probe materials. We report on attempts to answer those questions.

We attempted to measure the laser-input power at a wavelength of 355 nm that would damage the ends of sapphire fiber optics. We were surprised and pleased to learn that they survived an input power density of about  $3 \times 10^9$  W/cm<sup>2</sup>, which greatly exceeds the best that fused-silica fibers can do.

During a run of our new simulator to obtain an upgraded calibration curve for the improved YAG:Dy phosphors, we found that the phosphor appeared to form a eutectic, with the fused-silica cuvette used to hold the phosphor, when the temperature exceeded 1450°C. This result could have substantial ramifications in this and other high-temperature applications.

Our new proprietary detector package that replaced the original photomultiplier tube gave excellent results, with much better signal-to-noise ratio at a given temperature than the old package.

Our new plasma-spraying operation has succeeded in spraying YAG, which we think may be a technological breakthrough.

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## Introduction

FluoreScience, Inc. (FSI) is developing a probe to measure temperature in developmental slagging coal gasifiers. FSI is collaborating with faculty and graduate students from Tennessee Technological University (TTU) in this work. The temperature-measurement method uses thermographic phosphors (TPs) as the temperature sensors. The basis of the method and many of its applications are amply covered in the literature.<sup>1</sup> Reference 1 is a review article that includes references to other work.

The idea behind TP temperature measurements is conceptually straightforward. In practice, the method is complex. TPs are ceramics and similar materials that exhibit repeatable characteristics that are functions of temperature. One generates these characteristics by depositing the TPs on the surface whose temperature is to be measured, then subjecting the TPs to ultraviolet (UV) light. The resulting fluorescence, which exhibits the temperature-sensitive characteristics, is converted to an electrical signal by an appropriate photoelectronic detector. The electrical signal is directly related to the temperature. It is thus possible to build an instrument that measures temperature by using TPs as sensors.

For use in coal gasifiers, we have proposed using a probe with TP deposited on the inside of the tip. The probe would, like existing thermocouple probes, be inserted so that the probe tip projects into the interior of the gasifier. The biggest advantages of the TP probe would lie in the expected durability and low cost.

This third progress report covers further work intended to answer several crucial problems regarding the probe design and construction. One way to phrase these questions is as follows.

1. What numbers and/or conditions can we assign to the environmental parameters? The parameters include number and location of probes; type of materials used to construct the gasifier walls and their thermal characteristics; thickness of the walls; composition of the gases; and pressures, temperatures, etc.
2. Is there a suitable optimum ceramic material for the probe body? The ceramic will handle the stresses caused by temperature. It will be durable in the high-temperature-gas environment. It will sufficiently resist diffusion of high-pressure, hot gas such that a simple purge-gas technique can remove reactive gas from the interior.
3. Is there a satisfactory inexpensive method for coating TP durably onto the inside of the tip?

There are other crucial questions that we can address later, but these three could be “go/no-go” questions.

## Experimental

There are two basic strategies for getting the optical-excitation signal into the probe and getting the resulting fluorescence signal out of the probe. They are the clear-path method

(which uses lenses) and the fiber-optics method. Because of various unresolved mechanical, thermal, and optical uncertainties, it is by no means certain which of the two is superior. One of the principle issues involved in the fiber-optic method is how much laser power one can put into fiber optics without damaging the fibers. Our previous (unpublished) work using fused-silica fibers showed that they are definitely limited. With that in mind, we set up an experiment to measure the laser-damage threshold in sapphire fibers. We performed the experiment to measure how much UV power input from the laser is required to damage the sapphire fibers that we intend to use in the test-rig probe. By careful experiment design and layout, we were reasonably sure that almost all of a given (variable) power output would enter the face of the laser.

Our new experiment simulator allows us to simulate a wide range of field-experiment conditions in the laboratory. The new simulator has let us redesign our experiment setups so that the quality and accuracy of the data are dramatically improved over earlier versions. We used the simulator as a calibrator to obtain data for the best of the new batches of YAG:Dy that we received during the previous reporting period.

We compared the performance of our proprietary detector package with that of the photomultiplier in the calibration mode. We already knew that the new detector was far superior to the PMT in dynamic range and tolerance of blackbody background. However, we also knew that the new detector has a lower gain than the PMT package, which we thought might cause a problem in high-temperature small-signal situations in which too-small signals worsened the problem of poor signal-to-noise ratio.

We need to determine the effect of hot H<sub>2</sub>S on the phosphor. There exists the possibility that, at high enough temperatures, the H<sub>2</sub>S will chemically reduce the phosphor to its elemental form or to another compound, essentially destroying it for its intended purpose. The logistics of building an apparatus to test this hypothesis lie well beyond our capability, especially in terms of available funds. As this report is being written, we may have found a lead to an existing facility that can let us do a test.

The plasma-spraying work continued.

## Results and Discussion

To our great surprise, we found that the fiber passed or absorbed all of the input power that the laser could supply at 355 nm. This translates to about 20 MW peak power and an average power density into the fiber of about  $3 \times 10^9$  W/cm<sup>2</sup>. This is extraordinary, about a factor of 100 better power handling than the best fused-silica fibers. To the best of our knowledge, this has not been previously reported in the literature.

The results of the YAG:Dy calibration were very interesting. We were able to extend the calibration of YAG:Dy easily to 1450°C, whereas 1400°C was previously the upper limit, achieved with extreme effort. The goal is, of course, 1700°C. At 1450°C, we found another unexpected result. We have been using a cuvette made of fused silica to hold the phosphor powder. The phosphor melting point is above 2400°C and the fused-silica melting point is about 1800°C. However, just above 1450°C, the cuvette came apart

vigorously, scattering pieces all over the inside of the furnace. Visual examination of the pieces showed an apparent fusing of the glass and the phosphor into a new form. The most likely cause is the formation of a eutectic. Whatever the cause, we cannot go to a higher calibration temperature until we get a new container for the phosphor. We are trying to get a manufacturer of sapphire parts to supply a suitable substitute for the cuvette. None currently exists, so it will have to be prototyped, at typical prototype cost.

In comparing the performance of the new detector with that of the PMT, we were pleasantly surprised that the very substantial improvements (resulting from the optimized experiment design permitted by the simulator) gave such an excellent signal that the new detector greatly outperformed the PMT. We are now using our detector for all experiments. Nevertheless, we have ordered state-of-the-art voltage dividers for the PMT as well as a larger dynamic-range version of the tube, which we plan to compare with the new detector.

We got good first results from the plasma-spraying work. One of the main problems is in spraying YAG so that it remains YAG – and not some other crystal structure – after the spraying. We succeeded in that, which appears to be another technological first. The method will remain proprietary. The next step is to try to plasma-spray YAG with  $\text{Eu}_2\text{O}_3$  so that we get YAG:Dy, the desired phosphor.

Our negotiations with Delta Controls Corporation have paid off. They have agreed to supply several of their proprietary probe housings that we can use to test our concepts. Now we can build and test our probe concepts. We should be able to run them to maximum temperature in our simulator. Assuming we can actually use the test facility we mentioned in the Experimental section, we will also be able to test the phosphor/probe-housing combination in its intended environment, albeit at relatively low temperature.

## Conclusions

We have no new information regarding Question 1 of the Introduction. We continue to assume that the Texaco gasifier is typical. We know the number and location of the probes and the wall thickness. We have enough information about the composition of the gases, and of the pressures and temperatures, to proceed. We have a rough idea of the gasifier's wall materials. We know very little about the currently used probe's construction. However, we are now going to get to use proprietary probe housings from Delta Controls Corporation that are commercially successful in Claus –type reactors. These appear to represent the state of the art. Two features of delta's probe are especially attractive. First, their two-sheath design helps to prevent damage to the probe. The outer sheath connects to the steel shell of the reactor. The inner sheath (which contains the temperature sensor) connects to the refractory-brick lining. A half-inch clearance between the two sheaths allows some movement of the refractory relative to the shell without damage. Second, a nitrogen-purge system that allows indiffused acidic gases to be removed, with minimal damage to the temperature sensor. Note, though, that Pt/Pt-Rh thermocouples naturally “getter,” or attract and adsorb such gases, so their lifetime is limited even with purging. The attractiveness of the phosphor sensors is that they do not adsorb such gases.

We are still exploring the questions in (2) of the Introduction. We still have very little information. We are starting to suspect that no ceramic exists that will meet the temperature specification, the diffusivity specification, and the need to survive a slagging environment, but we are still looking vigorously. However, we have an idea that we intend to exploit using the Delta Controls Corporation probe housings. This idea, if successfully applied, will substantially the diffusivity of the probe ceramic.

With our new plasma-spraying results, we have made significant progress on answering Question 3 of the Introduction.

The finite-element modeling is still on hold until we find out how to determine the exterior loads placed on the probe by shifting of the refractory.

#### References

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<sup>1</sup> B. W. Noel, W. D. Turley, and S. W. Allison, "Thermographic-Phosphor Temperature Measurements: Commercial and Defense-Related Applications," Proc. 40<sup>th</sup> International Instrumentation Symposium (Instrument Society of America, 1994), pp. 271-288.