QUARTERLY
TECHNICAL PROGRESS REPORT
Report No. 5

Laser Ultrasonic Furnace Tube Coke Monitor
ERIP
Grant No. DE-FG36-98GO10302

For Reporting Period:
May 1, 1999 through August 1, 1999

Prepared For:
U.S. Department of Energy

Prepared By:
HARVEST ENERGY TECHNOLOGY
9253 Glenoaks Blvd.
Sun Valley, CA 91352

August 15, 1999
DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.
DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.
1.0 INTRODUCTION

This report summarizes the technical progress achieved during the fifth quarter of the ERIP project entitled, "Laser Ultrasonic Furnace Tube Coke Monitor." In Report No. 4, the development of an automated probe was described. The objective of work during this period was to enhance the sensitivity and signal to noise ratio of the probe measurement. Testing identified that the primary source of signal noise was traced to imperfections in the sacrificial stand-off, which was formed using a casting procedure. The stand-off transfers the actuation signal from the pulser to the surface to be measured and back to the receiver. The stand-off must be able to conduct the signal with a minimum amount of interference, while conforming to the shape of the target. Laminations, voids, and impurities contained in the casting result in attenuation and dispersion of the ultrasonic signal. Therefore, a significant effort was made to optimize the casting method for the production of the sacrificial stand-offs. This report describes the work performed to optimize the signal conductance of the sacrificial stand-off.

Project expenditures at the time of this report were approximately $68,000. These expenditures are consistent with the project progress.

2.0 BACKGROUND

The overall aim of the project is to demonstrate the performance and practical use of a probe for measuring the thickness of coke deposits located within the high temperature tubes of a thermal cracking furnace. This aim will be met by constructing a probe that will be tested using simulated coke deposits that are positioned inside of a bench-scale furnace. Successful development of the coke detector will provide industry with the only available method for on-line measurement of coke deposits.

3.0 PROJECT OBJECTIVES

A key project objective will be to verify that the coke detector can meet specific end-user criteria needed for successful commercialization of the device including the following:

1. Sensitive measurement of coke deposits ranging in thickness from 0-1/4"
2. Suitable for use on coils of 1 1/2" - 4" diameter
3. Suitable for use at coil temperatures up to 1050°C
4. Suitable for use on rough oxidized metal surfaces
5. Instrument reliability under practical operating environments
6. Measurement reproducibility
7. The instrument is operator safe
8. The probe can be used to make random measurements down the coil length at various access angles from available furnace ports
9. The probe can operate at stand-off distances of at least 10-20'
10. The instrument package can be assembled for less than $50,000-$75,000
4.0 PROJECT WORK

4.1 Sacrificial Stand-off Concept

Conventional ultrasonic transducers used to investigate the internal structure of materials can not be directly exposed to high temperatures. High temperature ceramic stand-offs can be used to thermally isolate the transducer from the hot surface. However, effective transmission of energy from the stand-off to the hot surface requires the use of a gel-like couplant. Unfortunately, these couplants cannot typically withstand temperatures above about 550 °C.

To overcome this problem, we have developed a stand-off composed of a fusible alloy that thermally isolates the transducer from the hot tube surface while providing the necessary coupling of the transducer to the measurement surface. Furthermore, the fusible alloy automatically conforms to rough, convex, tube surfaces, regardless of the angle of the measurement (angle of attack).

4.2 Optimization of Stand-off Production Method

The sacrificial standoffs were cast using a eutectic mixture of bismuth and tin. Figure 1 shows the mold and the fusible alloy stand-offs produced in the mold.

![Figure 1. Mold and stand-offs fabricated using fusible alloy](image)

In the initial casting procedure, the alloy was heated above its melting temperature and poured into a heated mold. The alloy and mold were then allowed to slowly cool to room temperature. However, because the mold bore contracts upon cooling and the particular alloy used for casting exhibits the anomalous behavior of expansion upon solidification, the solidified casting could not be easily removed from the mold.
A graphite lubricant was applied to the inside bore of the mold to assist in the removal of the casting, however this approach gave inconsistent results. To overcome the differential expansion problem, castings were made using an unheated mold into which superheated liquid metal was poured. This fast quench technique (Figure 2) was successful in minimizing the differential expansion between the cast alloy and the mold.

![Figure 2. Quick cast of the bismuth-tin sacrificial stand-off.](image)

However, the signal quality of the stand-offs produced using the fast quench technique was quite variable and generally much poorer than for the stand-offs produced by the slow cooling method. For instance, Figure 3 shows the signal emanating from a stand-off produced using the fast quench method compared to a signal emanating from a typical commercial ceramic stand-off.
Figure 3. Signal from stand-off using fast quench technique

Figure 4. Signal from a commercial ceramic stand-off
As with most liquid metals, the bismuth / tin alloy is susceptible to oxidation when heated to the molten metal state. The oxidation layer starts to form as soon as the molten metal surface is exposed to oxygen. As illustrated below in Figure 5, the oxidation layer is quite noticeable even after 1 minute.

![Metal surface initially exposed](image1.png) ![Metal surface at 1 minute exposure](image2.png)

**Figure 5.** Metal oxidation layer development

In the slow cooling casting method, the molten alloy is poured into the mold, and any oxide scale present in the casting floats to the surface before solidification occurs. However, in the fast quench casting method, the oxide impurities are trapped in the rapidly solidifying mold. These oxide impurities act as scattering centers for the ultrasonic signal, thereby resulting in a poor signal to noise ratio.

To overcome the problem of oxide contamination, a small vessel was fabricated that would deliver fresh metal from the bottom of the molten pool into the bottom of the stand-off mold, thereby virtually eliminating the oxidation exposure to the metal and carry over. The molten metal dispenser is illustrated in Figure 6.
With the metal oxide contamination minimized, virtually 100% of the stand-offs fabricated were of acceptable quality for both conductance and signal to noise ratio. Figure 7 illustrates signal from the alloy stand-off produced using the underflow dispenser.

Figure 6. Band-heated molten metal dispenser

Figure 7. Alloy stand-off produced using underflow liquid metal dispenser and fast quench casting method
4.3 Simulated Coke Layer Testing

To test the ability of the fusible alloy stand-off to distinguish the pulse echo from the coke layer of a simulated furnace tube, a 1/8 inch thick isostatically molded graphite slab was affixed onto a 3/8 inch Incoloy 800H plate. The dimensions of the graphite slab and the Incoloy plate were selected to be representative of the thickness of the coke and tube metal wall layers of a thermal cracking furnace about mid-way through the thermal cracking cycle. The test sample is shown in Figure 8.

![Figure 8. Side view of the graphite layer attached to an incoloy 800H base.](image)

The test sample was heated to a temperature of 350°F where upon the ultrasonic probe tip was activated and a measurement was recorded. As seen on the following page in Figure 9, the graphite peak is discernable coming just before the second incoloy reflection. For the purposes of measurement of the graphite layer, the first four reflections were averaged to calculate the thickness. The results of this initial test are given in Table 1.

The test results confirm the feasibility of measuring an internal coke layer using a manually operated probe that is contacted against a heated surface.
Figure 9. Incoloy Plate #2 oscilloscope results illustrating the incoloy and graphite signal reflections.
Table 1. Estimation of graphite layer thickness using sacrificial stand-off

<table>
<thead>
<tr>
<th>Incoloy Plate</th>
<th>Time (Divisions)</th>
<th>Delta (Divisions)</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.002 E-8 seconds / Division</td>
<td>182</td>
<td>177</td>
<td>177</td>
</tr>
<tr>
<td></td>
<td>359</td>
<td>177</td>
<td></td>
</tr>
<tr>
<td></td>
<td>536</td>
<td>177</td>
<td></td>
</tr>
<tr>
<td></td>
<td>713</td>
<td>177</td>
<td></td>
</tr>
<tr>
<td></td>
<td>890</td>
<td>177</td>
<td></td>
</tr>
</tbody>
</table>

Time x Average = 3.54 E-6 seconds

<table>
<thead>
<tr>
<th>Graphite Plate</th>
<th>Time (Divisions)</th>
<th>Delta Incoloy - Graphite</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.002 E-8 seconds / Division</td>
<td>322</td>
<td>140</td>
<td>139</td>
</tr>
<tr>
<td></td>
<td>498</td>
<td>139</td>
<td></td>
</tr>
<tr>
<td></td>
<td>675</td>
<td>139</td>
<td></td>
</tr>
<tr>
<td></td>
<td>852</td>
<td>139</td>
<td></td>
</tr>
</tbody>
</table>

Time x Average = 2.78 E-6 seconds

Speed = 2.55 mm/μs

Measured Thickness = 3.55 mm

Thickness Actual = 3.175 mm (1/8”)

% Error = 12%
5.0 PROJECT SCHEDULE AND BUDGET

The project expenditures to date are $68,000 or about 68% of the total allocated budget. The accomplishments of the program to date are as follows:

1. Development of a novel sacrificial stand-off that can be used for contact ultrasonic measurements at high temperature.
2. Optimization of a casting procedure for production of sacrificial stand-offs having a high signal to noise ratio.
3. Fabrication of an automated probe assembly.
5. Confirmation of the probe assembly for detecting the thickness of a simulated coke layer.

The work for the next period will involve further optimization of the probe measurement technique at moderate temperatures, and initial design of a water-cooled lance that will allow the probe to be inserted into a high temperature cavity to simulate probe use in a commercial furnace.