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Principal Author: Heng Ban (Associate Professor)

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Name and Address of Submitting Organization: University of Alabama at Birmingham
Department of Mechanical Engineering
1150 10th Avenue South
Birmingham, AL 35294-4461

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ABSTRACT

Advanced sensor technology is identified as a key component for advanced power systems for future energy plants that would have virtually no environmental impact. This project intends to develop a novel high temperature corrosion sensor and subsequent measurement system for advanced power systems. Fireside corrosion is the metal loss caused by chemical reactions on surfaces exposed to the combustion environment. Such corrosion is the leading mechanism for boiler tube failures and has emerged to be a significant concern for current and future energy plants due to the introduction of technologies targeting emissions reduction, efficiency improvement, or fuel/oxidant flexibility. Corrosion damage can lead to catastrophic equipment failure, explosions, and forced outages. Proper management of corrosion requires real-time indication of corrosion rate. However, short-term, on-line corrosion monitoring systems for fireside corrosion remain a technical challenge to date due to the extremely harsh combustion environment. The overall objective of this proposed project is to develop a technology for on-line corrosion monitoring based on a new concept. This report describes the initial results from the first-year effort of the three-year study that include laboratory development and experiment, and pilot combustor testing.

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INTRODUCTION

Background and Significance

Revolutionary sensor technology is a key component for DOE's approach for developing next generation energy plants that would have virtually no environmental impact. Relying on fossil fuels for a major share of our energy needs well into the 21st century and a diverse mix of energy resources, Vision 21 is the culmination of power and fuels research and development directed at resolving energy and environmental issues. The focus is to develop the critical technologies that underlie the components and subsystems ("modules") that are the building blocks of future energy plants. The key elements of the approach include: focusing on key technologies, stressing innovation and revolutionary improvements, producing early benefits, and emphasizing flexibility to meet market needs. One of the identified crosscutting technologies that are expected to be important, regardless of the actual configurations of future energy plants is the development of advanced sensors for highly integrated advanced energy systems.

Fireside corrosion is the external tube metal loss (wastage) caused by chemical reactions on water tubes exposed to the combustion environment in a furnace [1]. Corrosion is the leading mechanism for boiler tube failures [2, 3]. The direct economic cost of corrosion, through parts and labor to replace corroded equipment are often minor compared to the loss of production while the plant is under repair. For example, the cost of replaced power from the shutdown of a needed power plant can run into millions of dollars per day. Fireside corrosion typically occurs in high temperature, harsh combustion environment found in boilers and chemical recovery systems. The corrosion of boiler tubes can lead to the thinning of the tube reached more than 80% of the original thickness. Such excessive corrosion can lead to tube leakage or rupture, which can then lead to significant equipment damage and possible injuries to personnel, and in Kraft recovery boilers, smelt-water explosions.

Proper management and control of high temperature corrosion requires real-time information of corrosion rate [4]. Future trends in energy plant development tend to increase fireside corrosion due to introduction of technologies targeting emissions reduction, efficiency improvement, or fuel/oxidant flexibility. The availability of an on-line instrument capable of quantifying fireside corrosion rates would be a valuable new tool for plant operators who must meet environmental targets while minimizing the deterioration of valuable heat exchanger surfaces. Additionally, knowledge of localized corrosion rates provides critical information so that informed decisions can be made for maintenance and ongoing life extension of the plant [5-7].

This project attempts to develop a novel high temperature corrosion sensor and associated measurement system for advanced power systems. The novel sensor concept was conceived and examined in the Phase I study, and the intent of the proposed work is to develop a system based on the new concept and evaluate its feasibility at the laboratory and pilot scale. The investigation includes laboratory scale development of sensors, the probe

and the measurement system, followed by the evaluation and improvement of the system in the laboratory, and the eventual test of the complete system at a pulverized coal (PC) combustion furnace (1 MW) that has been demonstrated to have a fireside environment typical of that found in a PC-fired utility boiler. The focus of the proposed work is the design, fabrication and testing of the system that can function in a high temperature harsh combustion environment. The overall goal of the proposed research is to develop and prove the feasibility of the technology.

Current Knowledge and Technology

Current methods for corrosion measurement or monitoring fall into three main groups: downtime inspection, metal loss types, and electrochemical types [8-37]. The result of downtime inspection is of limited value for pro-active corrosion management because it provides only historical data. The simplest metal loss type is the weight-loss coupon, which is the most commonly used technique in corrosion research. A sample of the material of interest, of known weight, is exposed to the process for a known period. When it is removed, carefully cleaned and weighed, the change in weight is used to calculate the metal loss that may then be expressed as an annualized rate of loss (mils or millimeters per year). The coupon requires a relatively long exposure to the combustion process to yield accurate results. The constraints imposed by the time of exposure naturally limit the number of data points that can be obtained from a location, and ultimately do not detect process changes quickly.

Metal loss type sensors can be combined with electrical resistance measurement to provide an on-line monitoring capability. Because the electrical resistance of a current path increases as its cross sectional area is reduced, metal loss due to corrosion of the sample can be detected by an electrical resistance-measuring instrument. An Electrical Resistance (ER) sensor is often comprised of a sensing element that is basically a wire, strip or tube made of the alloy of concern, which is used to conduct the electric signal. When exposed to a corrosive environment, the cross-sectional area of the element is reduced. This increases the resistance of the sensing element, which can be measured and recorded as a function of time. Unlike electrochemical methods, ER sensors continue to function in the presence of non-conductive scales and are valuable tools for detecting under-deposit corrosion. As a simple and relatively inexpensive technique, ER is often the mainstay of a monitoring program in low-temperature applications, especially in the petroleum industry. In high-temperature combustion applications, however, ER sensors are significantly affected by instrumental and thermoelectric noise. The Electric Power Research Institute (EPRI) has made several attempts to develop the ER technique for fireside corrosion monitoring. Rowan Technologies of Manchester, United Kingdom (UK), developed a system. But the system failed to produce corrosion data in pilot scale testing. EPRI later sponsored the research team at the Southern Research Institute and the University of Alabama at Birmingham to develop the technology based on the lessons learned in the UK. The focus of effort is to minimize the persistent noise in the electrical measurement.

Electrochemical techniques measure the corrosivity of an environment independent of actual material loss. Linear Polarization Resistance (LPR) is the most widely used technique

of this type. It measures the DC current through the metal/fluid interface when the electrodes are polarized by a small electrical potential. As this current is related to the corrosion current, that in turn is directly proportional to corrosion rate, the method provides an instantaneous measurement of corrosion rate. This has advantages over metal loss methods, but is limited in the scope of its application by the requirement that the fluid be conductive, which in practice usually limits it to aqueous solutions. Other electrochemical techniques include Potentiostatic, Galvanostatic, Potentiodynamic, Galvanodynamic and AC Impedance Spectroscopy. None of these approaches have been successfully developed for field use as continuous monitors due to a variety of technical difficulties.

Electrochemical Noise (ECN) is a passive electrochemical technique that requires no polarizing current, but measures the naturally occurring electrochemical potential and current disturbances that result from corrosion. Electrical current noise is based on current variations between two nominally similar working electrodes, whereas potential noise is based on alterations between a working electrode and a stable, reference electrode. ECN is capable of giving accurate indications of general corrosion, pitting, and stress cracking when it is properly applied, but requires both expertise and complex data processing to be effective. Because ECN requires monitoring of very small signal fluctuations, this approach to corrosion monitoring is also affected by extraneous sources of signal noise in the plant. ECN is a relatively new technique and a company, Reaction Engineering International, has applied the technology in coal combustion application.

In summary, there is no mature technology for on-line fireside corrosion monitoring. Most efforts have tried to adapt existing technologies for low temperature applications to fireside applications. However, when adapted to fireside corrosion measurement, these technologies are either in development stage or significantly affected by errors or interferences inherent in the harsh combustion environment.

The New Concept

A new sensor concept was developed and examined in the Phase I study. The concept is based on a new measurement principle that has not been previously applied for corrosion measurement. The technique uses a metal-loss type approach, which involves exposing a sensing element to a corrosive environment and measure the thickness decrease of the element as a function of exposure time. As all online approaches to corrosion measurement, the challenge is to measure a thickness change of the order of 1 micrometer or less without destroying or removing the element from the corroding environment. The new concept converts the thickness measurement to area measurement. The technique employs thin-film coating of the material to be corroded on a substrate. The thickness of the coating varies continuously, for instance, from 0 to 40 micrometers over a length of 4 cm. When the sensor element is exposed to the corrosive environment and corrodes away a layer of a certain thickness, the decrease in thickness will be proportional to the coating area recession or decrease. Thus, the design converts the depth (thickness) change of 1 micrometer to a equivalent length change of 1 mm, which is much easier to determine.

The change in size or area of the metal coating can be measured on-line by electrical capacitance. A ceramic substrate with metal coatings on both sides is an electrical capacitor.

The capacitance is a function of the overlapping area of the metal coatings, and the thickness and dielectric properties of the ceramic. The change of capacitance is directly proportional to the change in the overlapping area of the metal coatings. The sensor design can include a front side coating area exposed to combustion, with linear thickness variation, and a backside coating not exposed to combustion. The decrease of coating area on the front side due to corrosion will be reflected proportionally by a decrease in capacitance.

The result of the Phase I study indicated that the new concept works in principle and is ready for further development. The Phase I study examined the fabrication materials and methods, coated the sensor elements using vacuum sputtering technology, tried the supporting electronic system for capacitance measurement and data acquisition, and measured the sensor response to oxidation in a muffle furnace. In addition, the study also measured dielectric constants of different substrates at different temperatures and characterized coating surface and structure using profilometry and Scanning Electron Microscopy. The Phase I examination of this innovative concept indicated that the concept can be further developed to quantify corrosion rate.

Technical Issues and Challenges

Although the new concept has shown promise, clear technical challenges remain. The obvious challenge, beyond the preliminary Phase I study, is to develop a system and prove its feasibility in laboratory, pilot combustor, and power plant testing. The technology has to be proven at a full-scale boiler before it can be developed into a commercial product. For corrosion monitoring in a combustion environment, proof-of-concept testing at the pilot scale is also an important step. Therefore, the following technical issues must be addressed to further develop the concepts and the technology.

(1) Options for Sensor Design and Fabrication.

We need to have answers to questions such as: what is the best design to achieve high sensitivity and low cost, or what is the best initial element thickness (or slope). For example, the element needs to be thick enough to have a long service life, yet also thin enough to provide a high signal-to-noise ratio. These answers will have to come from research and development at the laboratory scale and in the pilot combustor. The sensor designs need to take into account fabrication options. A compromise may have to be reached between an ideal design and practical requirements for fabrication. Each sensor will also have to be tested in coal-fired furnaces to see if they can survive and achieve the sensing objective.

(2) Complete Monitoring System Development and Improvement

The proof-of-concept step is to build a complete system and demonstrate its feasibility at pilot and full scales. There are many questions to be answered in designing and building the complete system, which needs to match the appropriate sensor designs from laboratory tests with robust probe design. A rugged, portable electronic measurement and control system that can function in the industrial environment need to be built. The measurement system also needs to automatically acquire and store data, and upload the data through (preferably) wireless communication.

(3) System Evaluation and Testing in a Coal Combustion Furnace

The corrosion monitoring system needs to be tested at both the pilot and full scales to reveal areas that require improvement or modification. In addition, the probe and ancillary instrumentation need to be tested for long-term performance to determine the service life of the sensor. Such tests will provide information on the stability and long-term performance of the new technology for fireside corrosion monitoring.

Although in theory the change in sensor capacitance is proportional to its change in thickness, the degree of deviation from the linear correlation has to be experimentally determined. Such an evaluation and the data resulting from this evaluation may provide a more accurate estimation of error range and confidence level of the corrosion rate determined by the ER method.

(4) Calibration of Results with Metrology Analysis

The corrosion rate determined from the novel sensor needs to be compared to true metal-loss measurement, either on the same element or on a separate weight-loss coupon. Industry accepts the amount of metal loss measured by coupon exposure as an absolute and correct measure of corrosion averaged over a period of time. Calibration of the corrosion rate measured by the new method provides confidence in the quantitative result from the sensor. Such comparisons will ultimately verify the result of the new sensor for fireside corrosion measurement and promote the acceptance of the technology in industry.

Anticipated Benefits

The novel sensor and supporting measurement system can be used in multiple end-use sectors to increase the overall efficiency of current and future energy plants. Having the availability of a real time fireside corrosion monitor can help to bring about one or more of the following: (1) quick diagnosis of corrosion problems; (2) monitoring the effect of operating condition changes on corrosion; (3) providing advance warning of system upsets that could lead to corrosion damage; (4) determining the need to invoke process controls; (5) establishing a realistic inspection or maintenance schedule; and (6) an accurate estimation of the useful service life of equipment. Because fireside corrosion has a large negative economic impact on energy plant availability, corrosion management can reduce such effects and increase the overall efficiency of the plant. The loss of electricity production due to plant downtime for repairing corroded waterwall tubes can run into millions of dollars per day. If the corrosion monitoring technology developed by this effort leads to the reduction of downtime for an average of one day each year for each boiler that is monitored, the overall efficiency increase for that boiler is a fraction of one percent. However, the total amount of saving could be in hundreds of millions per year due to the large number of units in the U.S. that would benefit from the application of this technology. In addition to economics, the effects of corrosion can also lead catastrophic explosions that endanger life and safety, which is an especially serious concern for Kraft recovery boilers in the pulp and paper industry. This research can help to reduce such risks by providing a continuous assessment of corrosion rates and can help operators determine the most efficient modes of operation. The increase in plant efficiency from corrosion management can directly reduce the emissions per

unit of production. Since corrosion is an inherent process for metals exposed to a chemically reactive environment, corrosion management and control is an important way to reduce its negative impact on plant availability for current and future energy systems.

This innovative concept has advantages over existing technological options and represents a significant departure from state-of-the-art approaches to corrosion monitoring technology. It can potentially result in a significant advancement in corrosion sensing technology. The ER method is still under development for combustion environments. ECN or other electrochemical techniques may present problems in interpreting the data when there is ash or slag deposition on the sensor element. An ash or slag layer on the sensor surface may change the electrochemical signal and compromise the quantitative capability of ECN method. In contrast, the new sensor is likely to be better suited to an environment where ash deposition and temperature fluctuation are common and almost unavoidable.

Research Objectives and Scope

The overall goal is to develop an on-line fireside corrosion technology based on an innovative concept. This project is to design and build a system and prove its feasibility at a coal combustor. The specific objectives are to:

- (1) Develop the sensor and electronic measurement system based on the new concept;
- (2) Evaluate and improve the system in a laboratory muffle furnace;
- (3) Evaluate and improve the system through tests conducted in a coal combustor.

The scope of work includes a comprehensive experimental program to be carried out in the laboratory and at a pilot-scale coal combustor. A corrosion monitoring system will be designed, tested, and improved in the laboratory and tested at a pilot-scale coal combustor. The experimental effort focuses on designing and building a complete system including the sensor, temperature controlled probe, and electronic measurement and data acquisition. The system will be tested and improved through evaluations using a laboratory muffle furnace. A pilot coal combustor (1 MW) will be used to evaluate the technology in a coal combustion environment. Such evaluation is crucial because many technologies fail during this stage of development due to rapidly varying combustion conditions, the aggressive industrial environment, and high levels of ambient electrical noise. The technology will be evaluated to ensure that (1) it works in a combustion environment, (2) the result can be confirmed by metal-loss measurements, and (3) the system is rugged enough for plant environment.

EXECUTIVE SUMMARY

Advanced sensor technology is identified as a key component for advanced power systems for future energy plants that would have virtually no environmental impact. This project intends to develop a novel high temperature corrosion sensor and associated measurement system for advanced power systems. Fireside corrosion should be properly managed with real-time corrosion rate information because it could lead to catastrophic equipment failure, explosions, and forced outages. However, short-term, on-line corrosion monitoring systems for fireside corrosion remain a technical challenge to date due to the extremely harsh combustion environment. The overall objective of this project is to develop a new technology for on-line corrosion monitoring based on a new concept by laboratory development and experiment, and pilot combustor testing. This report describes the progress in the first-year effort of the three-year project.

The specific objectives of the project are to: (1) develop the sensor and electronic measurement system based on the innovative concept, (2) evaluate and improve the system in a laboratory muffle furnace, (3) evaluate and improve the system through tests conducted in a pilot-scale coal combustor. The scope of work includes a comprehensive experimental program to be carried out in the laboratory and at a pilot scale combustor. The on-line corrosion monitoring system to be developed includes the sensor, temperature-controlled probe, and electronic measurement and data acquisition. The system will be tested and improved through evaluations using a laboratory muffle furnace. A pilot coal combustor (1 MW) will be used to evaluate and further improve the technology in a coal combustion environment. There are three main tasks in the project. The first task is to design and build a complete system during the first year. Task 2 is to evaluate and improve the system performance in a laboratory muffle furnace. The laboratory evaluation also includes establishing data analysis procedures and accumulating operation experience and prepares the system to be tested at the pilot coal combustor. Task 3 evaluates and improves the system in a coal combustor. The corrosion rates measured by the sensor will be compared with direct metal loss measurements of the sensor.

The task for the first year is completed, mainly the development of the probe and the measurement system in the laboratory. The completed work included the preparation and design of a corrosion probe, on which the corrosion sensor can be mounted. The first probe, which is slightly smaller in diameter, was redesigned with improvements to accommodate ceramic connectors for electrical connection. The probe temperature measurement and control was selected based on our experience, in addition to a probe temperature control system that was already available. The new controller is more portable and rugged, suitable for operation at ambient temperatures in a power plant environment. The probe temperature, or the temperature of the sensing element, is controlled with compressed air cooling. The electronic measurement with computer data acquisition was also developed in the laboratory. The data acquisition software was developed to allow the user to select data logging rate, the temperature for the sensor, and options for various measurement sequences. It can also automatically log and save the data for days or weeks without the need for operator

intervention. The probe temperature control was tested in laboratory muffle furnace to assess the control parameters and fluctuation range of the probe temperature.

In addition, more experience is gained at coal-fired power plants through the interaction with plant people. Such experience has a direct impact on the design of the sensor and measurement system, with the information on the practical constraints during the operation of the system in a plant environment.

The project progressed according the original plan and schedule. The next phase of the project is to examine the system in a laboratory furnace. It is expected that the next phase of the project should go well according the original plan.

With our experience in pilot scale furnace and power plant measurements, we may propose to add power plant trials in the final phase of the project. Power plant testing of the system developed in this project was not proposed in the original research plan. However, adding power plant tests to this project could accelerate the pace to bring the technology to practical application. Whether we can perform plant tests in this project will be further assessed in the future, when the system is proved in the laboratory and pilot furnace environment.

EXPERIMENTAL

Sensor Principle

The principle of the novel sensor is based on the electrical capacitance technique, which has never been used for corrosion measurement. The sensor consists of a ceramic substrate with metal coatings on both sides, which forms a classical electrical capacitor, as shown in Figure 1. The capacitance of the sensor is a function of the overlapping area of the metal coatings, the thickness of the ceramic substrate and the dielectric constant of the ceramic. The front side coating exposed for corrosion has a linearly varying thickness, or wedge-shaped. When the sensor element is exposed to the corrosive environment and corrodes away a layer of a certain thickness in wedge-shaped coating, this thickness decrease will be reflected on and proportional to the coating area reduction. This design converts very small change in the depth (or thickness) to substantial change in length, which is much easier to measure. Therefore, any loss of the metal by corrosion will result in a reduction of the coating area. The change in the area of the metal coating can be measured by electrical capacitance, which is directly proportional to the change of the overlapping area of the metal coatings. The thickness reduction due to corrosion is therefore quantified by the capacitance change. The corrosion rate can be determined based on the decrease in electrical capacitance over the time.

Sensor Design

Substrate Material Selection

The physical and chemical properties of the sensor substrate materials are important factors affecting the sensor design and performance. Since the sensor developed in this research will be used for corrosion monitoring in high temperatures and corrosive combustion environments, the substrate material needs to have properties of high thermal conductivity, low thermal expansion, good electrical insulation, corrosion-resistance and stability at high temperature environments.

The substrate of the sensor is a ceramic plate with high thermal conductivity and low thermal expansion coefficients. There are a few commonly used ceramic materials available for the sensor substrate. These materials include Beryllium Oxide (BeO), Aluminum Nitride (AlN) and Alumina (Al_2O_3). The comparisons of main properties of these materials are provided in Table 1 and Figure 2. Because of the presence of water vapor in combustion, AlN is not considered in this case.

Beryllium Oxide (BeO) is a ceramic material that combines excellent electrical insulating properties with high thermal conductivity. It is also corrosion resistant. Although beryllium oxide powders are toxic when inhaled or ingested, and the cost of machining is high, there are many applications that exploit its singular properties. BeO is a unique

material for electrical and mechanical applications, which require dielectric property, mechanical strength and high thermal conductivity. It is particularly well suited for a heat sink and heat dissipation medium in integrated circuitry.

Therefore, BeO is distinguished by thermal conductivity comparable to that of electrical conductors, while retaining dielectric constant, loss factor and dielectric strength in the range of the most efficient electrical insulators. This unique combination of properties in conjunction with good mechanical strength and thermal shock resistance enable BeO to be the best substrate material of the sensor in this research.

Aluminum Oxide (Al_2O_3) or Alumina is the second choice for the sensor substrate although its thermal conductivity is not as high as BeO. The high volume resistivity, chemical stability at aggressive and high temperature environments, high dielectric constant coupled with low dielectric loss and excellent electrical insulation lead to its wide applications in electronics as substrates. More importantly, Alumina costs much less than BeO.

Table 1: Properties for BeO and Alumina (99.6 % Al_2O_3)

Property	BeO	99.6% Al_2O_3
<i>Electrical</i>		
Dielectric constant @1 MHz	6.7	9.8
Dielectric loss @1 MHz	0.0002	0.0001
Dielectric Strength (KV/mm)	>9.5	35
Electrical resistivity (Ohm-cm)	> 10^{14}	> 10^{14}
<i>Mechanical</i>		
Density (g/cm^3)	2.88	3.75
Youngs Modulus (GPa)	340	390
<i>Thermal Properties</i>		
CTE($\times 10^{-6}/^\circ\text{C}$) (25~400 $^\circ\text{C}$)	6.7	6.9
Thermal Conductivity (W/Mk)	290	30

Because the dielectric constant of the sensor substrate is the usually a function of temperature and there are temperature fluctuations during the measurement. It is important to determine the relationship between the dielectric constant and temperature because the measured capacitance becomes temperature-dependent through the dielectric constant of the ceramic substrate. A change in sensor capacitance could be caused by two reasons, corrosion of the front coating or sensor temperature change. To eliminate the capacitance change from the sensor temperature variations and obtain the corrosion rate, a temperature compensation technique is used to process test data based on measured sensor temperature and the relationship between the temperature and dielectric constant. The compensation can remove the influence of temperature variations on capacitance measurement.

Sensor Fabrication

Sputtering Deposition Technology for Sensor Fabrication

The key for sensor fabrication is the forming a wedge-shaped front side coating to be exposed to combustion, with linear thickness variation. The sensor element design is shown in Figure 3. The backside coating not exposed to combustion has an equal thickness and is relatively easy to make. The coating materials are different for the two coating deposition. Since backside coating should have no corrosion, it is better to select a material that does not corrode in air environment.

Sputtering deposition is a technique by which atoms and ions of Argon or other gases from the plasma bombard a target, thereby knocking atoms off of the target. These material atoms travel to a substrate where they are deposited and form a thin film. It is necessary to provide a uniform and abundant supply of ions over the surface of the target. This is achieved by maintaining a charge differential facilitating the movement of the sputtered atoms across the gap between the target and the substrate.

DC magnetron sputtering deposition technology was used for one type of the sensor coating deposition. The fabrication included the design and machining of accessory parts for substrate installation during the coating, target material preparation and the coating fabrication. A Denton DVI-SJ-24 multi-cathode DC/RF magnetron sputtering deposition system, as shown in Figure 4, was utilized to deposit the iron coating on corrosion side and titanium coating on other side of the substrate. The DVI-SJ-24 is based on a “box coater” that provides easy access to substrates, sources, and instrumentation while maintaining excellent pumping characteristics. This system is designed to simplify the geometries necessary for the coordination of multiple source depositions. The system’s inherent flexibility allows for the operation of three sputter sources and the ability to heat, RF bias, and rotate the substrate. In a confocal cathode arrangement, the cathodes are focused on a central area of the substrate table. Table rotation during sputtering permits co-deposition, provides continuous substrate exposure to the cathodes, and results in excellent coating uniformity. With this arrangement, the cathodes can be smaller than the substrate and still provides uniformity of the coating.

We used a slow moving shutter for the specific purpose of depositing the linearly increasing thickness on the front side for corrosion. A special substrate holder with mask was designed and machined to support the sensor substrate during the sputtering. The substrate holder was adjustable in x, y, z directions to keep the mask and substrate in the appropriate position. Since the coatings on the two side of the sensor consisted of different target materials, the deposition rate and sputtering time were different for the coating on two sides. To achieve the required coating slope, experimental coatings were conducted to provide information on deposition rates. The needed exposure time of the substrate in plasma and shutter moving speed were calculated before the sputtering operation. The wedge-shaped coating was deposited by gradually moving the shutter over the coating area of the substrate during the sputtering. The area with longest exposure time had maximum coating thickness. The deposited coating had a gradual linearly changing thickness.

The low carbon steel 1010 was used as target materials to create wedge-shaped coatings. It took 7.5 hours for this coating deposition with total 45 steps along total length of

15 mm at 10-minute interval for each step. The maximum coating thickness measured by the profilometer was about 1.7 μm , because deposition rate for this material is very low.

For the backside coating, all unmasked area of the substrate had the same exposure time and the sensor holder rotated continuously during the sputtering. Since Titanium has a relatively higher deposition rate than low carbon steel 1010, the backside coating thickness achieved 2 μm during 1.5 hours of the deposition.

Plasma Spray Technology for Sensor Fabrication

Plasma spray process is basically the spraying of molten or heat softened material onto a surface to form a coating. Material in the form of powder is injected into a very high temperature plasma flame, where it is rapidly heated and accelerated to a high velocity. The hot material impacts on the substrate surface and rapidly cools down forming a coating. Such a plasma spray process carried out correctly is called a "cold process" (relative to the substrate material being coated) as the substrate temperature can be kept low during processing avoiding damage, metallurgical changes and distortion to the substrate material.

A plasma spray system in Plasma Process Inc., in Huntsville, Alabama was tested to fabricate the front and back side coatings. The front side wedge-shaped coating thickness varies from the thinnest to the thickest of 50 μm over a total length of 61mm. The backside coating has a uniform coating thickness of 100 μm . The target material was a low carbon steel "Atomet 95". In addition, a layer of Titanium coating was sprayed around the holes for electrical connection of the sensor. The purpose of Titanium was to prevent the coating oxidation (or corrosion) at electrical connection areas.

Probe Design

The probe for the application in pilot coal-fired furnace was re-designed to accommodate the ceramic connectors for the sensor element. The requirement for the probe includes mounting of the sensor element at the end, the temperature control of the sensor element with compressed air cooling, and the flange mount for insertion to the combustor. The existing probe is slightly shorter and smaller in diameter. The new design incorporated features for more ruggedness and future applications in power plants.

Probe Temperature Control and Data Acquisition

The temperature control system and the data acquisition system were selected. We previously used this temperature control system in pilot furnace experiments and had good experience with it. It was proved to be rugged and precise for the similar furnace probe applications. Our original temperature control system can control the probe temperature precisely. However the size of the control cabinet is large and not as convenient. Both systems would be evaluated in our experiments.

The computer data acquisition with a LCZ meter was developed. The 4-wire measurement technique was applied to measure capacitance. One pair of leads supplied test current to the device under test (DUT) and a separate pair of leads made the voltage measurement. The method can prevent the voltage drop in current carrying wires from affecting the voltage measurement. The 4-wire method were also arranged to eliminate the impedance from the electrical leads as source of error, and thus improved the measurement accuracy. The data can be converted to corrosion rate information on a continuous online basis. An OMEGA DPI8 thermometer connected to a thermocouple transfers the temperature of the sensor element to the computer. Data acquisition software was developed to perform automatic data collection by the computer to obtain the test data from the SR175LCR meter and OMEGA DPI8 thermometer respectively. The rate of data acquisition could be adjusted by program. The data acquisition computer could acquire and display the measured capacitance and temperature of the sensor on real time basis, and the measurement results were programmed to be automatically saved every four hours for further data processing and analysis. The data acquisition software can run continuously until it receives the stop command.

RESULTS AND DISCUSSION

The task for the first year is to design and build a corrosion probe and measurement system for the subsequent laboratory and pilot testing. This goal has been achieved. The completed work included the preparation and design of a second corrosion probe, in addition to the one already available for the laboratory and pilot experiments. The first probe is slightly smaller in diameter, whereas the redesigned second probe has improvements to accommodate ceramic connectors and longer length for potential power plant testing. The schematic diagram for the laboratory experiment setup is shown in Figure 5. The assembly drawing of the probe design is shown in Figure 6, and the component drawings are not shown. The wedge-shaped coating of the sensor element is shown in Figure 7.

The probe temperature measurement and control components were selected based on our experience in pilot and plant furnaces, shown in Figure 8. The existing probe temperature control system that was already available has a large cabinet, shown in Figure 9. The new controller is more portable and rugged as well, suitable for operation at ambient temperatures in a power plant environment. The probe temperature, or the temperature of the sensing element, is controlled with compressed air cooling.

The electronic measurement with computer data acquisition was also developed in the laboratory. The laboratory setup for the probe, temperature and measurement system is shown in Figure 10. A muffle furnace is used and the sensor end of the probe is inserted in the muffle furnace. The pressed air cooling, and temperature control system is connected. A desktop computer is used for data acquisition. But a laptop computer can also be used for the data acquisition and control. The data acquisition software was developed to allow the user to select data logging rate, the temperature for the sensor, and options for various measurement sequences. It can also automatically log and save data for days or weeks without the need for operator intervention.

In addition, more experience was obtained for the requirements in the plant environment for the corrosion measurement systems. We had meetings with plant people on such topics and toured plant facilities. Such experience has a direct impact on the design of the sensor and measurement system, with information on the practical constraints during the operation of the system in a plant environment.

Because our experiences in pilot scale furnace and power plant measurements, we may propose to add power plant trials in this project. Power plant testing of the system developed in this project was not included in the original research plan. However, power plant tests could accelerate the pace to bring the technology to practical application. Whether we can perform plant tests in this project will be assessed in the future, when the system is proved in the laboratory and pilot furnace environment.

The project progressed according the original plan and schedule. The next phase of the project is to examine the system in a laboratory furnace. It is expected that the next phase of the project should go well according the original plan.

CONCLUSIONS

The corrosion monitoring system was designed and built in the laboratory, meeting the target of the original schedule. More technical information regarding the performance of the system and further improvements will be studied in the second and third year of the project.

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FIGURES

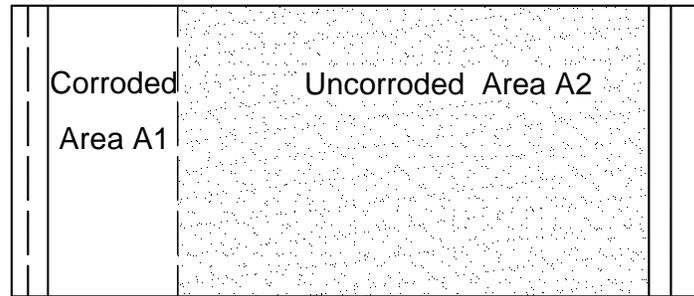
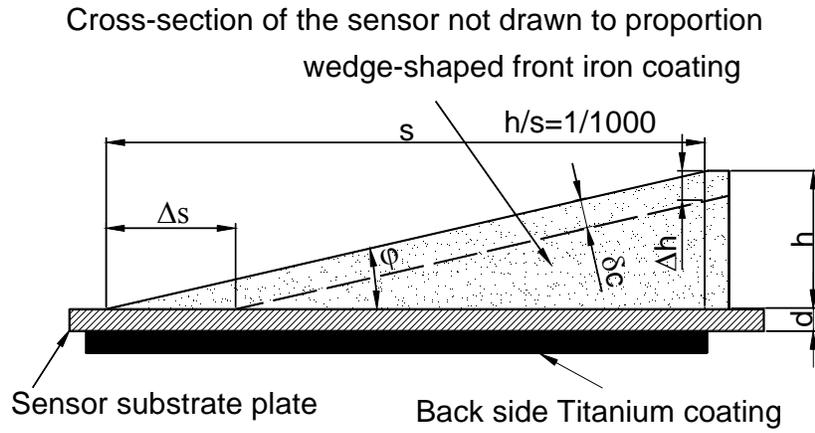


Figure 1 A schematic diagram of sensor principle

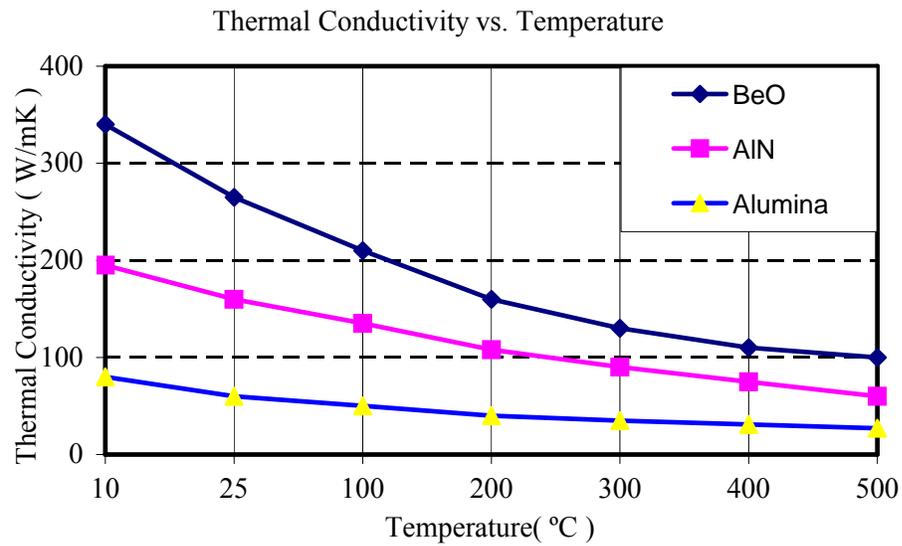


Figure 2 The thermal conductivity of BeO, Alumina (99.6%Al₂O₃) and AlN.

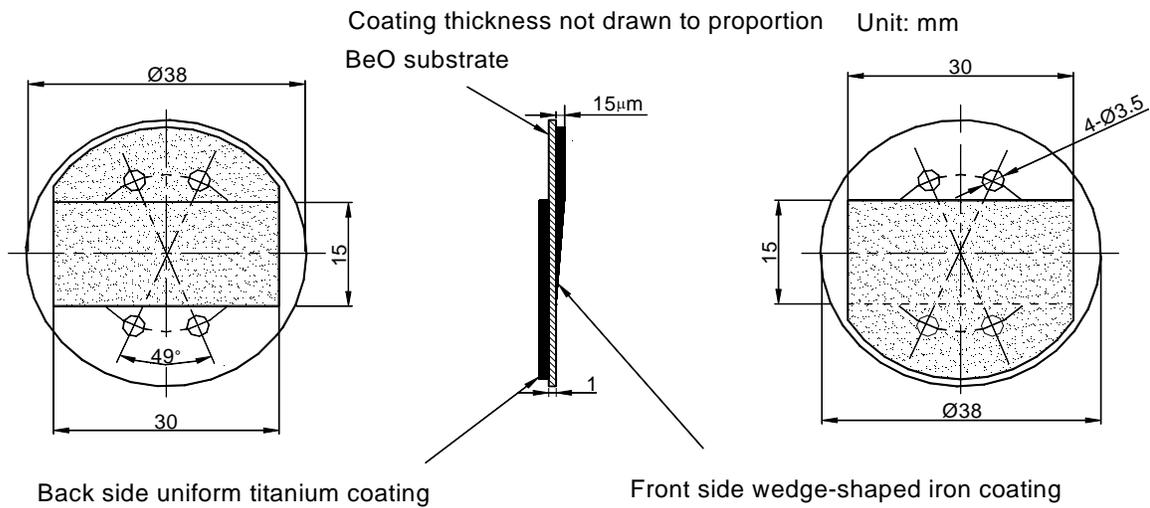


Figure 3 The drawings of the the sensor design.



Figure 4 A picture of the DC magnetron sputtering deposition system.

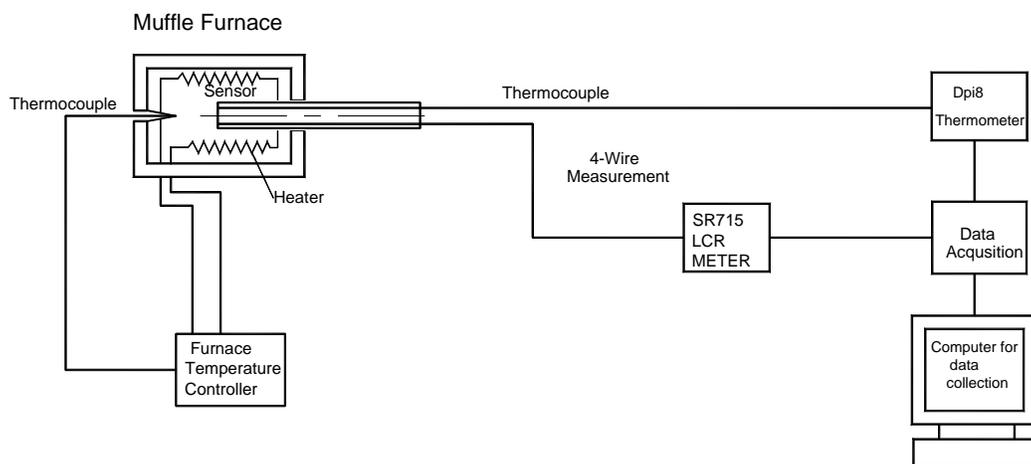


Figure 5 The schematic diagram for the laboratory experimental setup.

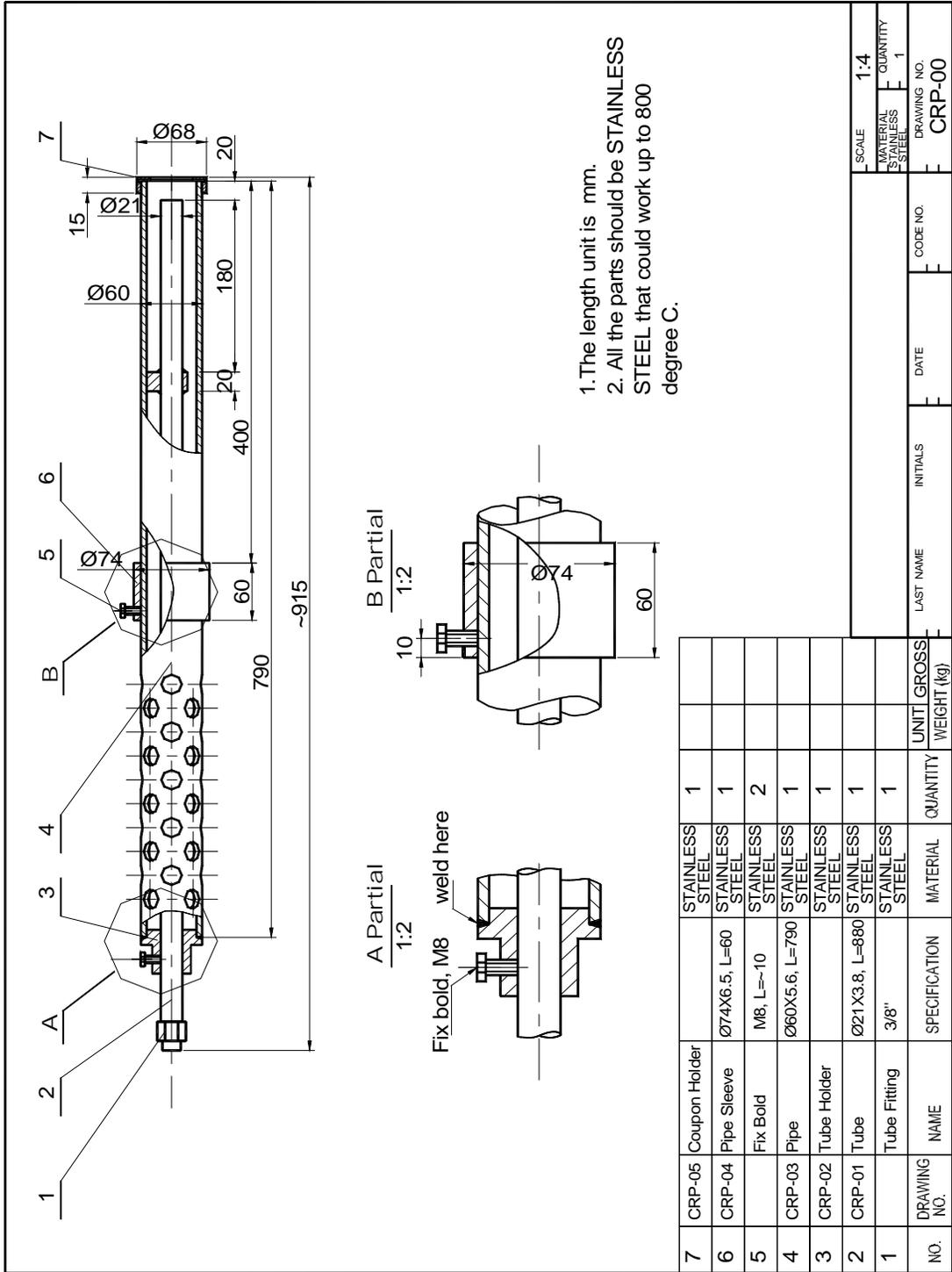


Figure 6 The assembly drawing of the probe design.



Figure 7 The pictures of wedge-shaped iron coating of one of the sensors.



Figure 8 The temperature control of the probe.



Figure 9 The first data acquisition and control enclosed in the cabinet.

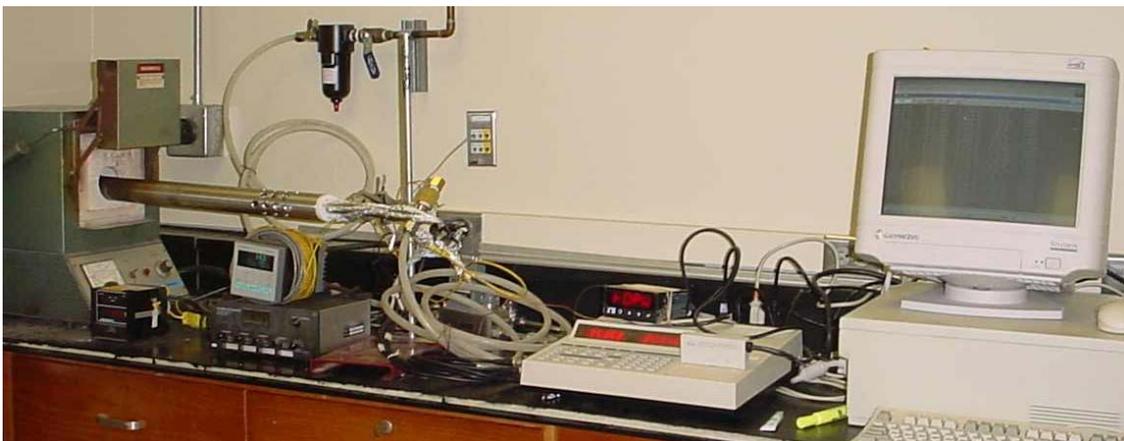


Figure 10 The probe and measurement system setup in the laboratory.