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ABSTRACT

Fireside corrosion in coal-fired power plants is a major obstacle to increase the overall efficiency for power producers. The increased use of opportunity fuels and low emission combustion modes have aggravated the corrosion on boiler tube walls in power plants. Corrosion-induced equipment failure could lead to catastrophic damage and inflict significant loss of production and cost for repair. Monitoring fireside corrosion in a reliable and timely manner can provide significant benefits to the plant operation. Current corrosion inspection and measurement are typically performed during scheduled maintenance outages, which is often after the damage is done. In the past, there have been many attempts to develop real time continuous corrosion monitoring technologies. However, there is still no short-term, on-line corrosion monitoring system commercially available for fireside corrosion to date due to the extremely harsh combustion environment.

This report describes the results of a laboratory feasibility study on the development effort of a novel sensor for on-line fireside corrosion monitoring. A novel sensor principle and thin-film technologies were employed in the corrosion sensor design and fabrication. The sensor and the measurement system were experimentally studied using laboratory muffle furnaces. The results indicated that an accurate measure of corrosion rate could be made with high sensitivity using the new sensor. The investigation proved the feasibility of the concept and demonstrated the sensor design, sensor fabrication, and measurement instrumentation at the laboratory scale. An uncertainty analysis of the measurement system was also performed to provide a basis for further improvement of the system for future pilot or full scale testing.

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INTRODUCTION

Background and Significance

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 the 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, a key technology for current and Vision 21 energy plants, requires a real-time indication 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].

Current Knowledge and Technology

Current methods for corrosion measurement or monitoring fall into three main groups: metal loss types, electrochemical types, and inspection [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 an 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 sensed 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, UK, developed a system but this system failed in pilot and full-scale testing. EPRI later sponsored the current research team (SRI and UAB) to develop a technology based on the lessons learned in the UK. This development effort is ongoing, with the focus on minimizing 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 uses 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 from low temperature to fireside applications. However, when adapted to fireside corrosion measurement, these technologies are either in their infancy or significantly affected by errors or interferences inherent in the harsh combustion environment. A new solution to this problem is needed.

Research Objectives and Scope

This research utilizes a new sensor principle based on a concept that can convert the thickness measurement to an area measurement. The area measurement is achieved by the measurement of the electrical capacitance. This sensor concept is new and there have been no reports or commercial products using such concept for corrosion measurement. The details of the sensor principle are to be described in the next section.

The overall goal of this investigation was to prove the feasibility of the concept by developing a novel sensor and the measurement system for short-term, on-line corrosion monitoring in combustion environments at laboratory scale. The sensor design and fabrication are based on thin-film deposition technologies. The specific objectives of the study were to:

- (1) study the feasibility of the sensor concept for on line corrosion monitoring by laboratory experiments;
- (2) examine the design and fabrication options for the sensor;
- (3) evaluate the influence of temperature on the measurement result;
- (4) establish the uncertainty and the sensitivity levels of sensor and the measurement system by both laboratory tests and analysis.

These objectives were achieved by laboratory experiments and analysis. An experimental system consisted of a muffle furnace, a LCZ meter, and computer data acquisition was established for the corrosion tests. High-sensitivity sensors were designed and fabricated based on the novel concept and thin film technologies. A four-wire measurement technique was used for the measurement of sensor capacitance. Corrosion experiments were conducted at the simulated combustion environments at different temperatures. The data were analyzed to show the relationship between the corrosion and the measured capacitance values. An uncertainty analysis was performed to evaluate the uncertainty of the overall system.

EXPERIMENTAL

Sensor Principle

The principle of the novel sensor in this research is based on the electrical capacitance (EC) technique. The sensor consists of a ceramic substrate with metal coatings on both sides, which forms a classical electrical capacitor, as shown in Fig. 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 arrangement equivalently 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.

Converting thickness measurement to area measurement is the essential part of the new method. For fireside corrosion sensors, the measurement often involves exposing an element to the corrosive environment and indirectly measuring thickness decrease of the element by analyzing change of electrical signal such as resistance. The most difficult task is to measure thickness change of the order of 1 μm or less in situ, i.e., without destroying or removing the sensor element from the furnace. In this new sensor design, the slope of the coating thickness can be very small, which magnifies the signal during the thickness to area conversion. Corresponding changes in capacitance can be easily established experimentally and theoretically for a wedge-shaped coating. The corrosion rate can be calculated theoretically using the capacitance change and sensor geometrical correlations indicated in Fig. 1 by the following equations (3)—(6).

$$\text{Coating area change: } \Delta s = \frac{\Delta C d}{\varepsilon} \quad (3)$$

$$\text{Angle: } \cos \phi = \frac{s}{\sqrt{s^2 + h^2}} \quad (4)$$

$$\text{Thickness decrease by corrosion: } \Delta h = \frac{\Delta s h}{s} \quad (5)$$

Thus, the corrosion rate R (thickness decrease by corrosion per hour) given by

$$R = \delta_c / \Delta t = \Delta h \cos \phi / \Delta t \quad (5)$$

$$R = \frac{\Delta C d}{\varepsilon} \frac{h}{\Delta t \sqrt{s^2 + h^2}} \cong \frac{\Delta C d}{\varepsilon} \frac{h}{\Delta t s} \quad (h \ll s) \quad (6)$$

where: s = the overlapping area per unit width of the sensor (mm^2).

h = the maximum thickness or height of the wedge-shaped coating (μm).

Δt = total exposure time exposed in corrosive environment at temperature T (hour).

d = the thickness of sensor substrate (mm).

ε = the dielectric constant of substrate at temperature T .

ΔC = the total capacitance change of the sensor due to corrosion (pF).

T = corrosion experimental temperature ($^{\circ}\text{C}$)

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 Fig. 2.

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 dielectricity, 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. The sensor using this substrate was shown in Fig. 3.

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. Its cost is much lower than BeO. The sensor using this substrate was indicated in Fig. 4.

Aluminum Nitride (AlN) is another ceramic material with high thermal conductivity and low coefficient of thermal expansion. The main drawback of AlN is its potential reactivity with water vapor in high temperature, moist environments. We looked into the issue and decided not to select AlN for our experiments for now.

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

Property	BeO	AlN	99.6% Al_2O_3
<i>Electrical</i>			
Dielectric constant @1 MHz	6.7	9.1	9.8
Dielectric loss @1 MHz	0.0002	0.0004	0.0001
Dielectric Strength (KV/mm)	>9.5	>15	35
Electrical resistivity (Ohm-cm)	> 10^{14}	> 10^{12}	> 10^{14}
<i>Mechanical</i>			
Density (g/cm^3)	2.88	3.27	3.75
Youngs Modulus (GPa)	340	350	390
<i>Thermal Properties</i>			
CTE($\times 10^{-6}/^\circ\text{C}$) (25~400 $^\circ\text{C}$)	6.7	4.7	6.9
Thermal Conductivity (W/Mk)	290	200	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.

Design Parameters

The novel sensor design was basically a plate capacitor with a wedge-shaped coating on one side and uniform coating on the other side. BeO and Alumina were selected for the sensor substrates. Two thin film technologies were applied for sensor fabrication. The 1st type of the sensor developed was indicated in Fig. 3 with substrate BeO. The coating was deposited by DC magnetron sputtering deposition technology. The thickness of the wedge-shaped front coating designed varied incrementally from 0 to 15 μm over a length of 15 mm with total overlapping area of 450 mm². The backside coating had a different material with uniform thickness. The size and circular shape was initially selected for convenience of installation on a probe tip.

The 2nd type of the sensor with Alumina substrate is shown in Fig. 4. The coating was fabricated by plasma spray technology. The overlapping area was about 6,100 mm². The shape and size the substrate was not particularly machined for probe installation. The purpose to develop these two types of sensors was to find an appropriate ceramic substrate material and cost-effective coating method to realize the ideal sensor design and its fabrication.

Sensor Fabrication

Sputtering Deposition Technology for Sensor Fabrication

The most difficult task in the sensor fabrication process is the requirement of depositing a wedge-shaped front side coating area to be exposed to combustion, with linear thickness variation. 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 the 1st type of the sensor coating deposition. The fabrication tasks 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 Fig. 5, 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 better than $\pm 5\%$ uniformity. Smaller targets also reduce cost of target material. Targets can also be configured for parallel sputtering to process large batches, if desired.

We used a slow moving shutter for the specific purpose of depositing the linearly increasing thickness on the front side for corrosion. A specific substrate holder with mask was designed and machined to support the sensor substrate during the sputtering, shown in Fig. 6. 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. In this research, 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. In this way, the deposited coating had a gradually 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 $1.7\text{ }\mu\text{m}$, because deposition rate for this material is very low. Figure 6 showed the coating deposition in progress.

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 $2\text{ }\mu\text{m}$ during 1.5 hours of the deposition. The coating deposition in progress was shown in Fig. 7.

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 as shown in Fig. 8. This 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.

The plasma spray gun comprises a copper anode and tungsten cathode, both of which are water-cooled. Plasma gas (argon, nitrogen, hydrogen, helium) flows around the cathode

and through the anode, which is shaped as a constricting nozzle. The plasma is initiated by a high voltage discharge, which causes localized ionization and a conductive path for a DC arc to form between cathode and anode. The resistance heating from the arc causes the gas to reach extreme temperatures, to dissociate and to ionize and form plasma. The plasma exits the anode nozzle as a free or neutral plasma flame, which is quite different to the Plasma Transferred Arc coating process where the arc extends to the surface to be coated. When the plasma is stabilized ready for spraying, the electric arc extends down the nozzle, instead of shorting out to the nearest edge of the anode nozzle. This stretching of the arc is due to a thermal pinch effect. Cold gas around the surface of the water-cooled anode nozzle being electrically non-conductive constricts the plasma arc, raising its temperature and velocity. Powder is fed into the plasma flame most commonly via an external powder port mounted near the anode nozzle exit. The powder is so rapidly heated and accelerated that spray distances can be in the order of 25 to 150 mm. Plasma spraying has the advantage that it can spray very high melting point materials. Plasma sprayed coatings are generally much denser, stronger and cleaner than other thermal spray processes.

A plasma spray system in Plasma Process Inc., in Huntsville, Alabama was used to fabricate the front and back side coating of the 2nd type of sensors in this research. The design of the sensors and the schematic diagram of the plasma spray process are shown in Figures 4 and 8. 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”. The overlapping area of the sensor is about 6,100 mm^2 . 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.

Experimental Setup

The measurement system in Figures 9 and 10 was designed and built for the corrosion test. The muffle furnace was maintained at a constant temperature by a temperature controller. Because the furnace temperature distribution and fluctuation were not completely uniform, an additional thermocouple was used to measure real time temperature of the sensor during the corrosion experiments. An OMEGA Dpi8 thermometer connected to a thermocouple transfers the temperature of the sensor to the computer. Data acquisition software was developed to perform automatic data collection by the computer to obtain the test data from the the SR175LCR meter and OMEGA Dpi8 thermometer respectively. The time interval 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 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. This experimental system is essentially a prototype for possible applications in pilot or full scale tests. The data can be converted to corrosion rate information on a continuous online basis.

Experimental Procedures and Data Analysis

Laboratory corrosion experiments were carried out with the fabricated sensors under different temperature at a muffle furnace. The main procedures for tests and analysis are described in detail below.

Room Temperature Test for System Performance

The complete system was assembled in the laboratory and evaluated at room temperature first before the formal corrosion experiments. The purpose was to investigate the stability of the measurement system, test the data acquisition software, and estimate the noise level and precision of the measurement signal. These preliminary experiments provided the sensitivity and accuracy of the measurement system. Improvements were made for the experiments and the data acquisition system. For example, the data acquisition software was programmed to automatically save the measurement data to a file without operator intervention to prevent unexpected data loss during long tests.

Dielectric Constant Measurement of Substrates

This measurement was to obtain the relationships between the temperature and the dielectric constant for BeO and Alumina substrates. The dielectric constant of the substrate is a key parameter affecting the sensor capacitance and is a function of temperature and test frequency. There is no published data in the literature on the temperature effect and with of the frequency range of our interest. Most material property tables simply give the dielectric constant of the substrates at 25°C at 1MHz.

Generally, the dielectric constant is used to determine the ability of an insulator to store electricity energy. The measured dielectric constant is the ratio of the capacitance induced by two metallic plates with an insulator. When a material is utilized in electric application where the high capacitance is needed, a high dielectric constant is required. The test can be done at different frequencies as required.

Both BeO and Alumina are ceramic materials with high dielectric constant, which are appropriate insulating materials for capacitors or sensors. The experiments were conducted using a plate capacitor and the existing measurement system by slowly increasing the test temperature from room temperature to the desired value. The dielectric constant of substrates with the elevated temperature was obtained from the test data.

Corrosion Tests in a Muffle Furnace

After the procedures and the measurement system were ready, the laboratory muffle furnace was used to simulate combustion environments for corrosion experiments at 200, 300, 350, 400, 600 and 650 °C. The corrosion experiments were repeated using different sensors and all data were processed to obtain the corrosion rates. These experimental results were compared with each other to verify the repeatability of the measurement. Muffle furnace tests also provided information on the stability and the performance of the system as a whole.

Data Analysis Procedures

Data analysis procedures were developed to convert the capacitance change of the sensor to the corresponding corrosion rate using the correlations derived above. According to the noise level and sensitivity of the measurement system, the minimum time to obtain statistically meaningful corrosion rate was determined accordingly. Methods and procedures for data averaging and reduction were investigated using standard spreadsheet software.

Based on the sensor principle, a theoretical model was established to examine the correlation between measured capacitance change and the corresponding coating thickness reduction of the sensor. This analytical model provided a basis for experimental data processing procedures for the corrosion tests and subsequent calculation of corrosion rates. Comparisons between the experimental results and theoretically calculated values provided confidence for the measured value of corrosion rate and prove the design concept.

Temperature compensation was applied to remove the capacitance change caused by temperature variations and get the actual amount of corrosion. Because the dielectric constant of sensor substrate material was temperature-dependent, the temperature variations inside furnace caused capacitance fluctuations. The data acquisition computer collected instantaneous capacitance value of the sensor during corrosion tests and the temperature of the sensor. Therefore, to get the actual corrosion rate, the measured capacitance of the element was compensated using Eq. (7). The compensated capacitance of the sensor element $C(T,t)$ at measurement temperature T at transient time t was calculated by:

$$C(T,t) = C(T',t) - dC/dT(T' - T) \quad (7)$$

where $C(T',t)$ is the real time sensor capacitance at time t collected by data acquisition computer during the test, $(T' - T)$ is the temperature variation from the nominal sensor temperature T . Usually, an accurate determination of dC/dT can be made either from the material property measured, or directly on the actual element in the experiments. The latter method has the advantage of being at the exact temperature of interest and based on the sensor itself. By comparing the temperature variations and the change in capacitance in short period, we can obtain the value dC/dT because the corrosion during the short period is

negligible. For example for the corrosion test data at 200°C, the value of dC/dT was calculated from the data shown in Fig. 11, which is 0.005 pF/°C. The corresponding time period for the data collection was about 30 seconds, which produces negligible corrosion. In fact, any value of dC/dT at other experimental temperatures can be obtained in the same way.

After compensating for temperature fluctuation, the decrease in measured capacitance reflected the amount of actual corrosion. The related change in coating thickness of the sensor with change in capacitance can be established experimentally and theoretically. Suppose total capacitance change of the sensor element after temperature compensation during the corrosion exposure time ($t-t_0$) is ΔC , which can be given by Eq. (8):

$$\Delta C = C(T, t) - C(T, t_0) \quad (8)$$

Thus, the corrosion rate R at a nominal test temperature T is given by Eq. (9):

$$R = \frac{\Delta C d h}{(t - t_0) \epsilon s} \quad (9)$$

An uncertainty analysis of the experimental system and the measured data was performed to provide the overall measurement uncertainty and assurance to the result. Furthermore, in order to obtain additional confidence in quantitative information provided by the sensor, the corroded elements were analyzed to determine the accuracy of the measurement. These methods included SEM examination and remaining thickness measurement with the profilometer and digital micrometer. These techniques allowed for a direct comparison between the corrosion rates integrated over test period and the actual amount of material lost to corrosion.

RESULTS AND DISCUSSION

Overall, both types of the sensors had real-time gradual capacitance decrease, indicating corrosion effect, at elevated temperatures during the tests. The corrosion was confirmed by other measurements. The sensors and the coatings showed good stability at temperatures up to 650 °C. The results demonstrated the feasibility of the sensor concept. Furthermore, the experimental results all showed the possible material candidates and fabrication methods for the novel sensor.

Dielectric Constant of Substrates

Two substrate materials, BeO and Alumina, were measured for dielectric constant properties. The correlations between the dielectric constant and the temperature are provided in Fig. 12. Experimental results indicated that the dielectric constant of these two materials was increased with elevated temperature. The dielectric constants of these two materials have no significant change from room temperature to 150°C. For BeO, the dielectric constant ϵ increases from 6.7 at 150°C to 7.9 at 450°C. The dielectric constant of Alumina is more sensitive to temperature, which increases from 9.3 at 150°C to 41.4 at 650 °C. The rate $d\epsilon/dT$ at a specific temperature increases faster as temperature rises. These data were used in the design process for the sensor (type 1) and later for calculating corrosion rates using temperature compensation.

Corrosion Test Results

A series of laboratory corrosion experiments were carried out using the muffle furnace and the fabricated sensors at different temperatures. The data shown below are all compensated for temperature fluctuation. To provide additional quantitative information and compare with the test result from the corrosion, each sensor was taken out before it was completely corroded. After the sensor cooled down, the remaining capacitance of the corroded sensor at room temperature was measured. The comparison of sensor capacitance before and after the experiment in room temperature provided additional confirmation on the percentage of the sensor coating was lost.

Experimental Results from the 1st Type of Sensors

Laboratory corrosion experiments using the 1st type of the fabricated sensors were performed at the temperature of 200, 300, 350 and 400°C, respectively. Figure 13 shows a picture of one sensor before and after test, which showed the corrosion gradually occurred in front wedge-shaped coating. Figure 14 shows that no corrosion occurred in the backside Titanium coating of this sensor. This ensured that the capacitance decrease of the sensor was

produced by metal loss of wedge-shaped coating in the front. During the corrosion tests, the temperature fluctuations were controlled within the range of $\pm 2^{\circ}\text{C}$ around the set value. Figures 16 – 19 showed that the capacitance decrease of the sensors and temperature fluctuations with exposure time. Thus, according to the capacitance reduction caused by corrosion, the average corrosion rates were calculated using Eq. [9] and provided in Table 2 below.

The experimental results in Figures 16 and 17 were obtained using the same one of the 1st type of the sensors for the corrosion experiments. The sensor was corroded initially at 200°C for 48 hours and then increased the temperature to 300°C for further corrosion of additional 6 hours. After cooling, the capacitance of the sensor was decreased by 50.4%, compared with the initial capacitance value before corrosion at room temperature. Meanwhile, using the calculated corrosion rates and integrating over corrosion exposure time, the percentage of capacitance reduction of the sensor is about 46.2%. The difference between the values may be caused by many factors including measurement uncertainty, temperature transitions, etc.

Similarly, Figures 18 and 19 shows the test results under 350°C and 400°C from another two of the 1st type of the sensors, respectively. The corrosion experiments were carried out at 350°C about 0.75 hours and 400°C about 0.5 hours. The total percentages of capacitance decrease were 79.1 % at 350°C and 67% at 400°C by measuring the difference before and after the test at room temperature. In addition, using the calculated corrosion rates, the same values derived from the integral method were 70.9 % for 350°C and 65% for 400°C respectively.

Experimental Results from the 2nd Type of Sensors

Corrosion tests for the 2nd type of the sensors were carried out at relatively higher temperature of 600°C and 650°C . The sensor was corroded for 24 hours first at 350°C , then 24 hours at 400°C and additional 24 hours at 500°C . No capacitance change was observed during each testing period, which indicated no reduction in overlapping areas for the sensor. Because the corrosion actually took place, this phenomenon indicated that the wedge-shaped coating had nonzero initial minimum thickness and this thickness was not completely removed during the first 72 hours. Thus, the furnace temperature was increased to 600 and 650°C to continue the corrosion experiment. After being stable at 600°C for about 1.5 hours, the initial minimum coating thickness was likely corroded and the capacitance began to decrease gradually, due to reduction of overlapping area. The experimental results were presented in Figures 20 and 21. The sensor capacitance was decreased by 18.6% based on the capacitance difference. Similarly, the calculated capacitance by integral method is about 16.7%. The average corrosion rates were provided in Table 2. These data showed a corrosion rate of 49 nm/hr at 600°C and relatively higher corrosion rate of 121 nm/hr at 650°C . Higher surface roughness in Figures 15(b) and 27 also provided evidence for the corrosion.

Table 2: Average corrosion rates at different test temperature

Sensors used for the corrosion tests	Corrosion Exposure Time (hours)	Temperature (°C)	Corrosion rate (nm/hr)
Type 1	48	200	1.98
	6	300	56
	0.75	350	1515
	0.5	400	1973
Type 2	42	600	49
	48	650	121

Post-exposure Analysis

Post-exposure analysis of the corroded sensor elements included SEM examinations and thickness measurement to assess the accuracy of the corrosion measurement. Quantitative comparisons between the corrosion rates indicated by the measured electrical capacitance approach and by direct physical measurement of the remaining thickness can provide valuable assurances that the sensor and the established measurement system was functioning properly.

The surface profilometer and digital micrometer were utilized to measure the coating thickness of both the non-corroded and corroded sensor elements. The results provided physical information that validated the electrical capacitance measurement. In this research, the surface stylus profilometer Alpha Step 500 was applied to measure the film coating thickness and substrate surface roughness of the sensors before and after the corrosion experiments. Data from remaining coating thickness examinations were applied to estimate how much the corrosion rate was.

Scanning Electronic Microscope (SEM) and optical electronic microscope were used to examine the coating microstructure of the sensors before and after the corrosion experiments. SEM and optical microscopic graphs can provide qualitative information about surface coating microstructure and the thickness and present physical information about the corrosion features of the sensors. Figures 22 and 23 shows the coating boundary and transition area of the one 1st type of the corroded sensor by SEM. The coating boundary and the transition area in pictures illustrated the corrosion near the substrate. Because the sample was not coated with a conductive layer for SEM analysis, Fig 23 does not have sufficient details to show the thickness difference between the exposed substrate and the remaining coating layer. Figures 24 and 25 show the microscopic picture of one 1st type of the sensors before corrosion test. Figures 26 and 27 indicate the microscopic pictures of the 2nd type of the sensors before and after corrosion. By comparison, microscopic examinations revealed surface roughness. It was observed from the pictures that the more prominent surface roughness or pitting was noted on the corroded sensors.

Uncertainty Analysis

Uncertainty analysis is the process of identifying and quantifying uncertainty during a measurement. It is quite important that engineers interpreting the results of the measurements have some methods for evaluating likely uncertainty. Measurement of physical quantities is always prone to measurement errors, which are grouped into two very general categories: bias error and precision error. Total error in any single measurement is the sum of bias and precision errors. Bias errors will shift the sample mean away from the true mean of the measured variable by a fixed amount. The bias error remains constant during a given series of measurement under fixed operation conditions. Calibration against a standard provides the most direct method to estimate bias, but the calibration cannot eliminate bias error, it can only reduce it. Precision errors will bring about a statistical distribution of measured values about the sample mean. When repeated measurements are made under nominally fixed operation conditions, precision errors will manifest themselves as a scatter of the measured data. Precision errors are identified and quantified through repeated measurements and a statistical analysis of the results. Since accuracy deals with the difference between the true value and the measured value, an accurate measurement has both small bias and precision errors. For multi-variable measurement, each variable contains same measure of uncertainty that will affect the result and propagate to the result. The propagation of uncertainty in the variables will yield an uncertainty estimate of the result. Thus, the measurement precision index and the bias limit are then combined to yield the uncertainty in the result.

Uncertainty Analysis for Corrosion Measurement Results

Uncertainty analysis of corrosion experiments was conducted to evaluate the measurement accuracy as well as the performance of the sensors and measurement instruments. Based on corrosion test results, the corrosion rate R , can be calculated by Eq. (10):

$$R = f(\Delta C, \varepsilon, d, h, s, t) = \frac{\Delta C d h}{\varepsilon t s} \quad (10)$$

Here, the corrosion rate R at specific test temperature is the function of the following variables: capacitance difference ΔC , dielectric constant ε , substrate thickness d , maximum coating thickness h , coating overlapping area s , corrosion exposure time t , and test temperature T . Since capacitance and dielectric constant were influenced by temperature T , the uncertainty of temperature T will propagate to ε , thus to the corrosion rate result R .

Based on the theory of uncertainty analysis for multi-variable measurements above, each measured value of the variables has its own uncertainty and the propagation of these uncertainties leads to an uncertainty in the corrosion rate R . Take measurement results of the variables in R obtained from the corrosion experimental data for uncertainty

analysis, the related statistical outcome can be provided for uncertainty analysis. So the mean value of the corrosion rate can be given by Eq. (11):

$$\bar{R} = \frac{\bar{\Delta C} \bar{d} \bar{h}}{\bar{\varepsilon} \bar{t} \bar{s}} \quad (11)$$

Consider both bias and precision errors for each variable, estimation of bias error relies heavily on the experience and on understanding of calibration accuracy and dimensional tolerances, precision error is given by the equation [38]. Thus, the bias and precision errors from each variable will propagate through to result and produce the result uncertainty. Table 3 provides the bias and precision uncertainty of the single variable in corrosion rate R, which are used to calculate the result uncertainty below.

Table 3: Bias and precision uncertainty of variables in corrosion rate R.

Viable x_i	Bias Uncertainty (B) $_{xi}/x_i$	Precision Uncertainty (P) $_{xi}/x_i$
ΔC	200°C	0.2%
	300°C	0.2%
	350°C	0.2%
	400°C	0.2%
	600°C	0.2%
	650°C	0.2%
d	1%	2%
h	1%	2%
s	1%	2%
ε	0.2%	1%
t	0.1%	0.1%

The uncertainty of the corrosion rate R can be estimated by equations (12)-(17)

$$P_R = \sqrt{\left[\frac{\partial R}{\partial \Delta C}(P)_{\Delta C}\right]^2 + \left[\frac{\partial R}{\partial \varepsilon}(P)_{\varepsilon}\right]^2 + \left[\frac{\partial R}{\partial d}(P)_d\right]^2 + \left[\frac{\partial R}{\partial h}(P)_h\right]^2 + \left[\frac{\partial R}{\partial s}(P)_s\right]^2 + \left[\frac{\partial R}{\partial t}(P)_t\right]^2} \quad (12)$$

$$B_R = \sqrt{\left[\frac{\partial R}{\partial \Delta C}(B)_{\Delta C}\right]^2 + \left[\frac{\partial R}{\partial \varepsilon}(B)_{\varepsilon}\right]^2 + \left[\frac{\partial R}{\partial d}(B)_d\right]^2 + \left[\frac{\partial R}{\partial h}(B)_h\right]^2 + \left[\frac{\partial R}{\partial s}(B)_s\right]^2 + \left[\frac{\partial R}{\partial t}(B)_t\right]^2} \quad (13)$$

The degree of freedom ν in the mean value of the corrosion rate is estimated from the Eq. (14)

$$\nu = \frac{\left\{ \sum_{i=1}^L \left[\theta_i \left(\frac{S_x}{\sqrt{n}} \right)_{x_i} \right]^2 \right\}^2}{\sum_{i=1}^L \left\{ \left[\theta_i \left(\frac{S_x}{\sqrt{n}} \right)_{x_i} \right]^4 / (n-1)_{x_i} \right\}} \quad (14)$$

The precision uncertainty P_{x_i} for x_i in Eq. (12) is given by Eq. (15)

$$P_{x_i} = t_{\nu,95} \frac{S_{x_i}}{\sqrt{n}} \quad (15)$$

Here, $L=6$, P_{x_i} represents precision errors of variables ΔC , d , h , s , ε and t in corrosion rate R , respectively, n represents the number of repeated measurement for variable x_i . According to value of ν , take 95 % confidence level, $t_{\nu, 95}$ can be determined by t-distribution from the table 3.6 [39] accordingly.

Thus, the uncertainty in the mean value of the corrosion rate R is estimated by

$$u_R = (B_R^2 + P_R^2)^{1/2} \quad (95\%) \quad (16)$$

The best estimate of the corrosion rate is given by Eq. (17):

$$R' = \bar{R} \pm u_R \quad (95\%) \quad (17)$$

Taking all corrosion experimental data of the sensors at each temperature for analysis, the corrosion rates with uncertainty were provided in Table 4 below.

Table 4: Corrosion rates with uncertainty at different temperatures.

Sensor Type	Temperature (°C)	Uncertainty $\frac{u_R}{\bar{R}}$ (%)	Corrosion Rate with Uncertainty R' (nm/hr) (95%)
Type 1	200	3.4%	1.98 ± 0.07
	300	3.5%	56 ± 2.0
	350	2.5%	1515 ± 38
	400	3.1%	1973 ± 62
Type 2	600	2.8%	49 ± 1.4

	650	3.2%	121 ± 3.9
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Sensitivity Analysis of Corrosion Measurement System

The purposes of the sensitivity analysis of the measurement system are to assess whether the sensor design and the measurement system are applicable for short-term on-line continuous corrosion monitoring. Data analysis for current laboratory experiments showed that the developed corrosion monitoring system achieved high sensitivity capable of detecting very small metal loss (0.4% of original maximum coating thickness) of the coating due to the corrosion. It was observed that the sensitivity of the system directly depends on the sensitivity of the fabricated sensors and the precision of instruments used. The sensitivity of the sensors was determined by the slope of the wedge-shaped coating exposed to corrosion environments and its thickness, sensor geometry such as the overlapping area, dielectric constant of substrate material, and chemical components of the target material for the coating as well as the coating deposition method. Understanding the relationship of these parameters with sensitivity is useful to optimize the sensor design and the measurement system.

Laboratory corrosion experiments demonstrated that an accurate measurement of the effective corrosion information was done in periods as short as in a few hours, which were illustrated in Figures 16-21 where the sensor capacitance reduced steadily and the corrosion rate was obtained in hours during the laboratory experiments. This proved that the sensor design concept and fabrication methods are applicable for the electrical capacitance-based (EC) sensor. Furthermore, experimental data analysis also showed that the minimum corrosion value to which the system can accurately measure is determined by measurement accuracy of LCR meter, and amounts to 0.4% of the sensor capacitance or the effective maximum thickness of the wedge-shaped coating. Thus, the laboratory corrosion results demonstrated that the novel sensor developed in this study was suitable for getting corrosion information in short term. The overall measurement system is highly sensitive to metal corrosion loss.

Comparison of Substrate Materials and Fabrication Methods

The ceramic BeO and Alumina are appropriate materials for the sensor substrates. The difference of the properties for these two materials lies in thermal conductivity and the dielectric constant, which are key parameters affecting the sensor design and its performance. BeO has higher thermal conductivity of 290 W/Mk and Alumina has higher dielectric constant as shown in Table 1. High thermal conductivity enables the sensor to dissipate heat quickly for the more uniform temperature distribution on the sensor. For same geometry and same flow rate, temperature control is easy for the sensor with substrate of BeO.

High dielectric constant of Alumina enables the sensor to have large capacitance for same sensor geometry, which can increase the sensitivity of the corrosion measurement. In addition, BeO is more expensive than Alumina and requires special equipment for

machining. Therefore, substrate selection for the sensor depends on the specific design and applications. It was proved that these two ceramic materials were applicable for the sensor substrates in this research.

Sputtering deposition and plasma spray technologies were tested for the fabrication of the sensor coating in this research. The challenge for the coating fabrication was to deposit a wedge-shaped coating on one side of the sensor substrate with thickness varying from 0 to the desired maximum thickness with a small slope. In order to find a cost-effective method for the coating fabrication, the above two methods were selected to fabricate type 1 and type 2 sensors, which were shown in Figures 13 and 15, respectively.

Surface morphology analysis demonstrated the coating by sputtering deposition is much smoother than the coating made by plasma spray deposition. Figures 24 and 26 shows the microscopic pictures of the sensor coating produced by these two methods, respectively. Comparisons of the pictures indicated that the coating roughness of the 1st type of the sensor was substantially smaller than that of the 2nd one. This is attributed to the difference of the fabrication methods. In sputtering deposition method, the metal atoms deposit to form coating on the substrate. In plasma spray method, the target material powder is heated and impinged to the substrate by plasma gas and cools down to form the coating. The sputtering deposition has relatively complicated system, low deposition rate and high cost for sensor fabrication.

Laboratory corrosion experiments demonstrated that the capacitance of the 1st type of sensors change quickly once the corrosion experiments started. The reason is that the coating thickness is lower and the coating corrodes away before a layer of oxide protection scale is formed. Meanwhile, the measurement results from the 2nd type of the sensor have no capacitance reduction during first 72 hours although corrosion actually took place. This phenomenon proved that the wedge-shaped coating had nonzero initial minimum thickness. To make the sensor capacitance decrease, this initial layer has to be removed. The time to acquire accurate corrosion information depends on the size of the initial minimum thickness and corrosion monitoring conditions.

Quantitative comparisons between the corrosion results from the two types of sensors provided valuable information about coating methods. All sensor coatings were of good quality and no coating peeled off during corrosion experiments. The sensors were in accordance with the design concept and functioned properly to provide corrosion monitoring in high temperature combustion environment. In conclusion, both the sputtering deposition and plasma spray technology are suitable for coating fabrication in this research.

Future Work

Future work of this project is to improve and develop a system suitable for short-term, on-line corrosion monitoring at pilot scale. While substantial progress has been achieved through laboratory testing, actual utility service exposures are necessary because a number of important environment parameters are impossible to simulate at laboratory. One of the main future tasks is to develop the corrosion probe shown in Fig. 28 with associated electronics mounted, and integrates the probe into the corrosion measurement and process

control system. The single sensor element can be cooled to monitor corrosion at any desired temperature. The temperature of the corrosion elements can be controlled to resolution of 1 °C or better and the capacitance of the corrosion element can be directly temperature compensated in software.

The probe can be made of a stainless tube with an internal air-cooling supply tube and electrical connections and convenient to allow easy sensor installation, removal, quick electrical connection and air hose connections. This probe can be exposed to actual coal-fired boilers for corrosion monitoring to investigate the potential of this novel system for on-line short-term corrosion monitoring in pilot scale power plants.

CONCLUSIONS

The corrosion studies have demonstrated that the sensor and measurement system are suitable for short-term, on-line corrosion monitoring in laboratory experiments.. Corrosion experimental results in a muffle furnace agreed with the trends established in theoretical design concept of the sensors and the results are reliable. The actual utility service exposure in field testing is a natural following step required to further verify the conclusions drawn from laboratory corrosion experiments because a lot of environmental parameters cannot be completely simulated in the laboratory, such as actual deposits on the sensor, large temperature gradients within the ash deposits, and flue gas temperature fluctuations inside the boilers.

In summary, the following conclusions can be drawn from current laboratory corrosion experiments:

1. Laboratory proof-of-concept corrosion experiments showed the feasibility of the novel sensor concept. Sensors had high signal-to-noise ratio and could detect slight changes of coating thickness change in the muffle furnace tests.
2. Design and fabrication options of the novel sensors were demonstrated. The sensors survived high temperature environments for corrosion monitoring. The fabricated sensors have met the theoretical design requirements.
3. Special method and tools were developed for the Magnetron sputtering deposition and plasma spray methods to produce a coating of wedge shape on sensor substrates. The methods were proved to be able to perform as designed.
4. The electronic measurement system assembled for laboratory experiments proved to have the capability to provide corrosion information quickly under different experimental temperatures. The minimum time required for a measurement of corrosion rate depends on sensor type, coating material and thickness as well as coating method.
5. Uncertainty analysis indicated that the measurement uncertainty was within 4% of the measured mean value. Sensitivity analysis of the experimental system estimated the performance of the sensor and corrosion measurement system, which can lead to optimize the sensor design and fabrication method as well as the measurement system in future.
6. It is anticipated that the future development of this system is to integrate air-cooling capability to control the sensor temperature for pilot scale testing in power plants boilers and incorporate the system into plant control system for pilot scale corrosion monitoring

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FIGURES

Fig. 1 The schematic diagram of the sensor design principle

(a)

(b)

Fig. 2 Thermal properties of the ceramic materials: BeO, Alumina (99.6%Al₂O₃) and AlN, (a) thermal conductivity and (b) linear thermal expansion.

Fig. 3 The drawing of the 1st type of the sensor designed in this research

Fig. 4 The drawing of the 2nd type of the sensor designed in this research

Fig. 5 View of DC magnetron sputtering deposition system used in this research

Fig. 6 A picture showing sputtering coating deposition in progress for fabrication of the sensor wedge-shaped coating.

Fig. 7 A picture showing sputtering coating deposition in progress for fabrication of the sensor backside coating.

Fig. 8 Schematic diagram of the plasma spray process.

Fig. 9 Schematic diagram of the corrosion measurement and data acquisition system

Fig. 10 Picture of the corrosion measurement and data acquisition system
Fm

Fig. 11 Typical value of dC/dT measured from small temperature fluctuations under 200°C, which is used to calculate corrosion rate at this temperature.

Fig. 12 Dielectric constant change of substrate material with increased temperature (a) for BeO and (b) for Alumina (92%Al₂O₃).

(b)

Fig. 13 Pictures of wedge-shaped iron coating of the 1st type of the sensor (a) before corrosion and (b) after corrosion.

Fig. 14 Pictures of back side Titanium coating of 1st type of the sensor (a) before corrosion and (b) after corrosion.

Fig. 15 Pictures of wedge-shaped iron coating of 2nd type of the sensor (a) before corrosion and (b) after corrosion

(b)

Fig. 16 Corrosion test results under 200°C (a) sensor capacitance change and (b) sensor temperature fluctuation during the corrosion test (sensor type 1 used).

Fig. 17 Corrosion test results under 300°C (a) sensor capacitance change and (b) sensor temperature fluctuation during the corrosion test (sensor type 1 used).

(a)

(b)

Fig. 18 Corrosion test results under 350°C (a) sensor capacitance change and (b) sensor temperature fluctuation during the corrosion test (sensor type 1 used).

Fig. 19 Corrosion test results under 400°C (a) sensor capacitance change and (b) sensor temperature fluctuation during the corrosion test (sensor type 1 used).

Fig. 20 Corrosion test results under 600°C (a) sensor capacitance change and (b) sensor temperature fluctuation during the corrosion test (sensor type 2 used).

(a)

(b)

Fig. 21 Corrosion test results under 650°C (a) sensor capacitance change and (b) sensor temperature fluctuation during the corrosion test (sensor type 2 used).

Fig. 22 SEM photomicrograph of sputtered iron coating boundary of the corroded sensor 200x (sensor type 1 used).

Fig. 23 SEM photomicrograph of sputtered iron coating transition area of the corroded sensor surface 200x (sensor type 1 used)

Fig. 24 A microscopic picture of the 1st type of the sensor coating fabricated by sputtering deposition technology before corrosion 500x.

Fig. 25 A microscopic picture of the 1st type of the sensor coating fabricated by sputtering deposition technology after corrosion 500x.

Fig. 26 A microscopic picture of the 2nd type of the sensor coating fabricated by plasma spray technology before corrosion 250x.

Fig. 27 A microscopic picture of the 2nd type of type the sensor coating fabricated by plasma spray technology after corrosion 500x.

Fig. 28 Probe for pilot scale corrosion test.