CONSTRUCTION AND TESTING OF A FLUE-GAS CORROSION PROBE

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J. I. Federer and J. A. McEvers

ABSTRACT

The selection of suitable materials for industrial, waste-heat-recovery systems requires assessment of corrosion of materials in various flue-gas environments. Such assessments involve exposing candidate materials to high-temperature flue gases and analyzing the effects of the exposure conditions. Because corrosion is related to flue-gas chemical composition and temperature, variations in temperature complicate the determination of corrosion rates and corrosion mechanisms. Conversely, a relatively constant temperature allows a more accurate determination of the effects of exposure conditions. For this reason, controlled-temperature flue-gas corrosion probes were constructed and tested for exposure tests of materials. A prototype probe consisted of a silicon carbide tube specimen, supporting hardware, and instrumentation for controlling temperature by internal heating and cooling. An advanced probe included other tubular specimens. Testing of the probes in an industrial-type furnace at a nominal flue-gas temperature of 1200°C revealed that temperature control was inadequate. The cooling mode imposed a substantial axial-temperature gradient on the specimens; while the heating mode imposed a smaller gradient, the heating capacity was very limited. The probe, as currently designed, is not a cost-effective method for obtaining corrosion data in high-temperature industrial-furnace flue gases. Other approaches are suggested, and details of construction and testing of a prototype probe and an advanced probe are presented.

INTRODUCTION

Metallic heat exchangers are used extensively to recover heat from flue gases of industrial furnaces. Typically, heat is transferred from the flue gases through a partition wall to air or steam. The preheated air can be used for combustion in the same furnace, thereby decreasing fuel use.

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The temperature of the metallic partition wall is limited to about 850°C because of severe oxidation or other corrosion and loss of strength that occurs at higher temperatures. Preheated combustion air temperatures, therefore, range from 500 to 800°C. A heat exchanger with ceramic tubes, providing the partition wall can tolerate much higher temperatures (>1200°C), can produce combustion air preheated to higher temperatures (1000 to 1100°C), and, therefore, can save substantially more energy than a metallic heat exchanger.

The U.S. Department of Energy, Office of Industrial Technologies [formerly Office of Industrial Programs (DOE-OIP)], has conducted research and development of high-temperature, ceramic heat exchangers through industrial contractors in recent years.^{1,2} A common problem in heat-exchanger concepts is the durability of ceramic materials exposed to corrosive, high-temperature flue gases in certain industrial applications. Conventional exposure tests in industrial-furnace flue-gas streams have frequently been used to assess the corrosion behavior of candidate materials for critical components such as heat-exchanger tubes, headers, and structural members.³⁻⁶ These tests involve installing characterized specimens of various ceramic materials in a furnace, recovering the specimens at a later date, and analyzing the specimens to determine the extent of corrosion or other degradation. The disadvantages of this type of test include:

- Specimen temperature is not controlled and sometimes not well known.
- Flue-gas temperature cycles frequently in some furnaces; therefore, specimen temperature also cycles.
- Corrosion data are based on a complicated temperature-time history.
- Corrosion rates for specific temperatures are not determined.
- The effect of temperature on corrosion rates is not determined.
- Extrapolation of data to other furnaces with different temperature-time histories is difficult.
- Stresses caused by atypical thermal cycling can cause additional degradation.

A possible solution to these problems is a ceramic corrosion probe with temperature-control capability provided by auxiliary heating and cooling. The probe would consist of one or more materials exposed to

fluctuating temperatures in furnace flue gases. Auxiliary heating or cooling within the probe would allow control of the probe temperature within a wide range of flue-gas temperatures. Several probes could be operated simultaneously at temperatures of interest in a combustion environment. Either constant temperatures or selected variations representing a particular heat exchanger could be maintained. Knowledge of the temperature-time history would be useful in subsequent interpretation of corrosion results. The probe concept is not new; metallic corrosion and fouling probes have been widely used.^{7,8} Usually cylindrical pipes, they are cooled by air flowing through the bore with strategically located thermocouples to indicate temperatures. Because these probes were designed to operate below the flue-gas temperature, auxiliary heating was not required.

As shown in Table 1, disadvantages of a corrosion probe include the limited number of specimens and the cost per specimen relative to conventional exposure tests. Conventional exposure tests could still be used to screen the corrosion behavior of a large number of materials. Then, the

Table 1. Comparison of corrosion test methods

Criteria	Conventional	Probe
Materials per test	Many	Few
Cost per specimen	Low	Moderate
Temperature known	Maybe	Yes
Temperature controlled	No	Yes
Temperature-time history	Complicated	Simple
Corrosion rate at temperature of interest	Maybe	Yes
Effect of temperature on corrosion rate	No	Yes
Data that can be extrapolated	Maybe	Yes
Furnace shutdown required for installation and removal	Yes	No

corrosion probe could be used to obtain pertinent data on the better materials. Table 1 indicates that probes would be advantageous in other respects, especially in determining corrosion rate at a particular temperature or as a function of temperature. The construction and testing of a prototype probe and an advanced probe are described herein.

PROTOTYPE PROBE

Designed by using a computer model to evaluate the effects of size, cooling air-flow rate, heating capacity, and flue-gas temperature and mass-flow rate on the range of temperature control (see the Appendix),^{9,10} a prototype probe was constructed for testing in an industrial furnace. The probe consists of a tubular ceramic (SiC) specimen, a stainless steel end fixture, and instrumentation for controlling temperature (Fig. 1). Air

Fig. 1. Schematic of corrosion probe.

flowing through the bore provides cooling. A double-spiral SiC electric heating element was initially tried as a heat source in the probe, but gas combustion in the bore was found to be superior in terms of maximum temperature and temperature uniformity. A microprocessor-based instrument* accomplishes temperature control. This device measures the probe temperature by means of an attached thermocouple and computes an appropriate output signal to drive the proportional pneumatic air and fuel-gas flow control valves via the current-to-pressure (I/P) converters. The output from the process controller is a 4- to 20-mA signal, with a value of 12 mA used as a control "null" point. A higher signal opens both the air and fuel-gas control valves, while a lower signal allows only cooling air to flow. Gas combustion in the probe is initiated by a high-voltage discharge in the end fixture at the inlet end of the SiC tube. A 3000-V dc power supply with suitable current-limiting resistors continuously generates the spark discharge. Figure 2 shows the prototype probe being test-fired in the laboratory.

A Hexoloy SA SiC[†] tube with nominal measurements of 25.4 mm OD x 19.1 mm ID x 91 cm long formed the specimen portion of the probe. This material was selected because it possesses many of the properties needed for heat-exchanger applications, e.g., high-temperature strength, oxidation resistance, thermal shock resistance, and availability in the form of tubes. Most DOE-sponsored ceramic heat exchanger projects in recent years have selected a silicon carbide ceramic for the primary heat-transfer element. Two type-S thermocouples in oval, double-bore alumina insulators were attached to the outer surface of the SiC tube at locations measuring about 30 cm from each end. The insulators were secured to the tube at several locations with platinum wire covered with an alumina cement. One thermocouple connected to the temperature controller served as the control sensor, while the other was connected to a temperature recorder. The axial distance between the thermocouples was about 30 cm.

*570 Series, Barber-Colman Company, Loves Park, Ill.

†Carborundum Company, Niagara Falls, N.Y.

Fig. 2. Prototype probe being heated by internal gas combustion in the laboratory.

ADVANCED PROBE

The method of thermocouple attachment on the prototype probe was inadequate, and the use of only one test material (SiC) was undesirable. Both deficiencies were addressed in the advanced probe, shown schematically in Fig. 3. The SiC tube is retained both as a specimen and as a support for other ceramic specimens. The tubular ceramic specimens are attached to the SiC core tube by means of a cement. Each specimen has a longitudinal slot to accommodate one or more thermocouples in double-bore insulators (Fig. 4). The slot width at the outer surface of the specimen is slightly less than the large diameter of the oval insulator; therefore, the insulator is captured in the slot and is secured further by an alumina cement. The alumina cement in the annulus between specimen and core tube contains 50 wt % SiC powder. The SiC powder serves as an inert filler and decreases the amount of shrinkage during sintering of the cement. The powder may also increase the thermal conductivity of the cement, but this possibility was not determined by measurement.

Fig. 3. Schematic of advanced corrosion probe with specimens of different materials.

Fig. 4. Schematic of assembly of a specimen onto the SiC core tube.

Two alumina cements were investigated for this application. One cement, designated AC1010^{*} for this study, contained approximately 92% Al_2O_3 and 7% SiO_2 ; the other, designated KT95,[†] contained approximately 84% Al_2O_3 and 14% CaO . Slotted specimens of Al_2O_3 and mullite were attached to SiC core tubes with each cement alone and with each cement containing 25 and 50 wt % SiC powder. The specimens were thermally cycled ten times between 20 and 1300°C at a heating rate of about 220°C/min. The Al_2O_3 specimen attached with KT95-50 wt % SiC broke by axial cracking, and cracks were present in the KT95 cement in the other two specimens. No cracks or other evidence of degradation were evident in the specimens attached with AC1010 and AC1010-SiC powder mixtures; therefore, the AC1010-50 wt % SiC mixture was selected for attaching specimens onto the SiC core tube of the advanced probe.

Several oxide ceramics were selected as specimens for the advanced probe: Al_2O_3 , mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), ZrO_2 stabilized with Y_2O_3 [$\text{ZrO}_2(\text{Y}_2\text{O}_3)$], and SiC whisker-toughened Al_2O_3 ($\text{Al}_2\text{O}_3\text{-SiCw}$). Previous industrial-furnace exposure tests had showed that these four materials have

^{*}Alfa Products, Morton Thiokol, Inc., Danvers, Mass.

[†]Babcock & Wilcox Company, Augusta, Ga.

better resistance than silicon-based ceramics to combustion atmospheres containing alkali compounds.^{5,6} Their corrosion behavior, therefore, could be compared to that of the SA SiC core tube, which, being exposed to the flue gases in several regions, formed a fifth specimen material. The oxides were in the form of tubes with inner diameters ranging from 26.6 to 30.9 mm as compared to the 24.9-mm OD of the SiC core tube. Therefore, the annulus to be filled with cement varied from about 0.85 to 3 mm in thickness, depending on the specimen. Rubber O-rings or strips of masking tape 3 to 4 mm wide were used as temporary spacers to center the specimens on the SiC tube while filling the annulus through the slots in the specimens. The cement, mixed with water to prepare a thick paste, hardened sufficiently in about 1 h to allow the spacers to be removed. After the four specimens and two thermocouples were cemented in place, the probe was heated to 1260°C and held for 2 h to sinter the cement. Figure 5 shows the advanced probe before testing in the ORNL steam plant furnace. A type-S thermocouple designated T1 was located about 6 mm from the outer end of the zirconia specimen, and another thermocouple designated T2 was located about 6 mm from the inner end of the mullite specimen, a separation distance of about 27 mm. T1 and T2 also designate the temperatures indicated by these thermocouples.

As shown in Table 2, the outer diameters of the oxide specimens ranged from 30.6 to 35.6 mm, and the lengths were 50.8 or 52.6 mm. Table 2 also shows that the coefficients of thermal expansion (CTE) of the oxide ceramics range from 5.5×10^{-6} to $10.5 \times 10^{-6}/^{\circ}\text{C}$. Because the CTE of SiC is about $5 \times 10^{-6}/^{\circ}\text{C}$, each of the specimens will expand more than SiC. During heating of the SiC tube by internal-gas combustion, the temperature of the specimens will lag behind that of the tube because heat must be transferred across the cement-filled annulus. The presence of SiC powder in the cement might enhance the thermal conductivity of the cement and minimize the temperature lag. However, the possibility exists that rapid heating and expansion of the SiC tube by internal-gas combustion would stress the oxide specimens, especially mullite.

Preparation of the advanced probe required considerable effort and expense. The cost of components, materials, and labor for machining, assembly, and installation at the test site is estimated to be \$25,000.

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Fig. 5. Advanced probe before testing.

Table 2. Dimensions and CTEs of oxide specimens

Material	Outer diameter (mm)	Inner diameter (mm)	Length (mm)	CTE ^a (in./in.°C)
Al ₂ O ₃	34.3	28.3	50.8	8.5E-06
Mullite	32.7	27.6	50.8	5.5E-06
ZrO ₂ (Y ₂ O ₃)	35.6	30.9	52.6	10.5E-06
Al ₂ O ₃ -SiCw	30.6	26.6	50.8	7.5E-06

^aAverage value for the range 25–1000°C except 25–500°C for Al₂O₃-SiCw material.

PROBE TESTING

A heating test of the prototype probe was conducted in the laboratory using propane and oxygen. Propane was used because natural gas was not available for the probe at the ORNL steam plant, where the probe was to be tested later; presumably, natural gas could be used as well as propane. Attempts to obtain a stable flame in the bore with propane-air mixtures were mostly unsuccessful because the flame would be quenched or transported to the exit end of the SiC tube by the combustion air. However, ignition was easily achieved near the inlet end of the SiC tube with a propane-oxygen mixture. A probe temperature of about 1000°C was obtained in the laboratory (Fig. 2) with average flow rates of about 27 and 135 L/min (1.2 and 6.8 mol/min) of propane and oxygen, respectively. Some air was also drawn into the mixture by the gas supply tube. The prototype probe was then installed in a boiler firebox at the ORNL steam plant, which burned coal during an exposure period of about 6 weeks. This test aimed to determine the capability of the system to control temperature while the probe was exposed to flue gases ranging in temperature from approximately 950 to 1200°C. Without auxiliary heating or cooling, the probe rapidly reached temperature equilibrium with the flue-gas temperature of 1200°C. Then, cooling air was forced through the probe to control the temperature at a value below the flue-gas temperature. With an air-flow rate of about 163 L/min (5.8 cfm), the minimum temperatures were about 1100 and 1200°C for the inlet end and outlet end (30 cm apart) thermocouples, respectively,

indicating that temperature equilibrium was attained between the probe and the cooling air during passage through the bore. Subsequent calculations suggested that the cooling air would attain the temperature of the probe at about the midpoint for an air-flow rate of ~283 L/min and that the location of temperature equilibrium moved closer to the inlet end for lower flow rates.

Unfortunately, the safety approvals required for burning gas in the bore of the probe could not be completed until the probe had been in the furnace for about 5 weeks. By that time both thermocouples had failed, and the furnace had been temporarily shut down. Before withdrawing the probe, however, the gas-ignition system was tested and found to be operating satisfactorily. The probe, when removed, was covered with thick deposits of ash and slag from the coal combustion environment (Fig. 6). The cement used to attach the thermocouples had spalled, and the thermocouple wires had reacted with the deposits. The method of thermocouple attachment obviously had to be improved for successful use of the probe. The SiC tube wall also exhibited a surface recession of 0.3 to 0.4 mm in the hottest part (midpoint to outer end) on the upstream side, indicating the corrosive nature of the environment.

When the advanced probe was installed, the furnace was initially heated with natural gas so that testing could be conducted in the absence of fouling deposits; however, the maximum temperature attainable at the probe location was only about 400°C. After performing some tests at that temperature, the furnace was heated to about 1200°C with coal for further testing. After only 24 h the probe had accumulated ash and slag deposits similar to those shown in Fig. 6.

In an actual application the instrumentation would be required to maintain the probe at a constant temperature as the furnace temperature changed. Control would be accomplished by cooling or heating as required. An objective of the tests was to determine the range of temperature control with the available cooling and heating capacities. Figure 7 shows the results of a cooling test at a nominal furnace temperature of 400°C. The initial temperature T_2 was about 30°C lower than T_1 because thermocouple T_1 was located deeper into the furnace. (T_1 was about 6 mm from the outer end of the zirconia specimen, and T_2 was about 6 mm from the inner end of the

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Fig. 6. Prototype probe after exposure to coal combustion products.

Fig. 7. Results of a cooling test at a nominal furnace temperature of 400°C.

mullite specimen.) The probe temperature decreased about 200°C as the flow rate of air increased to about 350 L/min. The final T2 was about 40°C lower than T1; therefore, cooling increased the axial-temperature difference between the specimens represented by T1 and T2.

The opposite effect occurred in T1 and T2 during a heating test (Fig. 8). As the set-point (SP) temperature advanced, the control system initially applied cooling air, then it proceeded into the heating mode. Within a few minutes the cooling air-flow rate decreased to zero, and gas combustion occurred. During the next few minutes, temperature T2 exceeded T1. When T1 reached the set point, T2 was about 30°C higher. As in the case of the cooling test, T2 was affected more than T1, and, again, an axial-temperature gradient was imposed on the specimens. The reason for this behavior, of course, is that the temperature of either cooling air or gas-combustion products increases as the gases proceed through the bore. When the air or combustion products reach T1, their temperature has changed from the value at T2 because of a tendency towards thermal equilibration with the probe.

Heat-exchanger tubes being used to preheat air typically operate at a temperature about 200°C below the flue-gas temperature. Therefore, the

Fig. 8. Results of a heating test at a nominal furnace temperature of 400°C.

corrosion probe would often be required to simulate heat-exchanger tube exposure, that is, operate in the cooling mode. Unfortunately, at a nominal furnace temperature of 1000 to 1100°C the effects of cooling and heating on axial-temperature gradients were even greater than at 400°C. Figure 9 shows that increasing cooling increased the axial-temperature gradient between T1 and T2. (The upturn in temperature at the highest flow rates was caused by a fluctuation in furnace temperature.) These results show that operating the probe in the cooling mode imposes a substantial axial-temperature gradient on the specimens and that the greater the amount of cooling, the larger the gradient. On the other hand, when the probe was operated in the heating mode to raise the temperature above the flue-gas temperature, the heating capacity was very limited (Fig. 10). At a nominal furnace temperature of 1100°C, heating affected T2 more than T1 as was the case at a furnace temperature of 400°C. T1 increased only 50°C and never reached the set point during the heating test, while T2 gradually increased to the set point. This small heating effect consumed oxygen at a rate of ≈ 2 mol/min, a rate that would consume a standard size cylinder of oxygen containing ≈ 5000 L (at STP) in ≈ 2 h. During this test the gas mixture was substoichiometric with respect to oxygen: if more oxygen had been used, the

Fig. 9. Results of a cooling test at a nominal furnace temperature of 1000°C.

Fig. 10. Results of a heating test at a nominal furnace temperature of 1100°C.

heating effect would have been larger, but the oxygen consumption rate would have been even higher. Therefore the cost of heating a probe by internal-gas combustion is likely to be prohibitively expensive.

The results of testing the probe in an industrial-type furnace can only be interpreted as unsatisfactory for the intended purpose (Figs. 7-10). The limited heating capacity by internal-gas combustion required an extremely large amount of oxygen. Alternatively, internal air cooling caused a substantial axial-temperature gradient. The latter effect was anticipated qualitatively, but not to the extent actually experienced. A furnace with fluctuating temperatures would require varying amounts of cooling air, which would cause the temperature of the specimens to vary accordingly. The tests showed that neither the heating nor the cooling capacity could significantly affect T_1 , the thermocouple farthest from the inlet end. When the costs of the temperature-control equipment, construction of the probe, and monitoring the probe during an exposure are balanced against the data obtained from a probe with such poor temperature control, we must conclude that a fully controlled probe is not cost-effective.

The temperatures T_1 and T_2 would be approximately the same at very low and very high flow rates of cooling air. At very low flow rates, $T_1 \approx T_2 \approx T_G$, where T_G is the flue-gas temperature. This is a case of essentially no control. At very high flow rates $T_2 = T_1 \ll T_G$, a case where both temperatures are forced very low simply to make them approximately the same. The latter case may not be useful for corrosion testing because the resulting low temperatures are not representative of heat exchangers. The position of specimens along the length of the probe affects their temperature, and their spacing affects the axial-temperature gradient. We have determined by testing that these problems are too complex to be resolved in the relatively simple flue-gas corrosion probe.

ALTERNATE APPROACHES

If the corrosion probe is not cost-effective, how should high-temperature corrosion data be obtained? The noncontrolled method mentioned early in this report, i.e., installation of a large number of specimens of

different materials in several industrial furnaces, should be used to separate the obviously better materials from the group. The better candidates should be further evaluated in terms of factors such as ease of fabrication, strength, thermal shock resistance, and cost to select only a few viable candidates for heat-exchanger materials. Then, tubular probes, each consisting of a single material like the previously discussed prototype probe, would be prepared. Using one material per probe would eliminate the cost of machining and attaching slotted specimens, as was done for the advanced probe; however, a method of thermocouple attachment would still have to be devised. Several such probes, equipped with thermocouples and provided with controlled internal cooling (no internal heating), would be installed in an industrial furnace. Cooling the tubular probes would cause axial-temperature gradients, thereby simulating the condition in heat-exchanger tubes. Following an exposure, the extent of corrosion of the probes would be determined. One disadvantage of this approach is that separate temperature-control systems would be required for each material, because a probe would consist of only one material; the previously discussed advanced probe consisted of several materials. Thus, the cost of obtaining detailed corrosion data by this conservative approach would be substantial.

An alternate, less conservative approach would utilize the corrosion results of numerous specimens in several different furnaces to identify a few viable candidates, as before. Final selection would omit the use of probes described above and rely on physical and mechanical properties and cost and availability (the method used in previous DOE-sponsored heat-exchanger projects). Surveillance specimens of the same material as the heat-exchanger tubes could be installed in the heat exchanger for periodic retrieval and examination, or actual tubes could be periodically removed for examination. In this manner, the progress of corrosion under actual heat-exchanger conditions could be assessed. This approach carries more risk because of less extensive testing but would be less expensive.

CONCLUSIONS

The work that has been performed supports the following conclusions.

1. Either internal heating or cooling imposes an axial-temperature gradient on the probe and on any set of specimens arranged along the axis of the probe. This precludes using a probe with multiple specimens unless different temperatures are desired; however, achieving the desired temperature in a particular specimen would probably not be predictable without some experience with this type of probe.

2. Internal heating by gas combustion requires oxygen for reliable ignition and a stable flame, but the cost of oxygen for extended heating would be prohibitive.

3. Internal heating by gas combustion was minimal and could not significantly affect the heat balance with the furnace; therefore, this type of auxiliary heating should not be used.

4. Alternate approaches to the corrosion probe include (a) exposure tests of numerous materials without temperature control to identify the better materials within a group; (b) further investigation of corrosion of a selected, limited number of materials in controlled cooling probes that simulate temperature gradients in heat-exchanger tubes; and (c) surveillance specimens installed in an actual heat exchanger to monitor corrosion during use. Options (a) and (b) would involve the highest cost and the lowest risk, while (a) and (c) would involve the lowest cost and higher risk.

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phases of testing was greatly appreciated. Finally, we appreciate D. L. Balltrip for preparing the final manuscript.

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APPENDIX

DESIGN OF A CONTROLLED TEMPERATURE FLUE GAS CORROSION PROBE*

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ABSTRACT

The need to determine suitable materials for use in industrial waste heat recovery systems requires assessment of materials degradation in various flue gas environments. Such an assessment generally involves exposing candidate materials to the environments, then analyzing the effects of the exposure conditions. Degradation of materials is related to flue gas chemical composition, temperature, and thermal cycling behavior. Temperature variations, especially atypical thermal cycling complicate the task of characterizing such phenomena as corrosion rates and corrosion mechanisms. A relatively constant temperature would allow a more accurate determination of the effects of flue gas chemical composition and temperature. In response to this need, a flue gas corrosion probe has been designed for exposure tests of materials. The probe allows materials to be tested at nominally constant temperatures within a wide span of either constant or changing flue gas temperatures. This paper describes the design and fabrication of the corrosion probe system.

INTRODUCTION

Exposure tests in industrial furnaces have been used in recent years to compare the high-temperature corrosion of various candidate materials for heat exchangers. Specimens have been placed in the flues of aluminum remelt furnaces (Russell et al., 1983, Federer et al., 1985, and Federer and Jones, 1985), steel soaking pits and steel reheat furnaces (Federer et al., 1985), and a glass furnace (Weber and Tennery, 1979). In most cases specimens were exposed to ambient furnace conditions for a time period determined by the furnace production schedule. Specimens were installed during a furnace shutdown, and were removed during a subsequent shutdown. During the exposure period temperatures sometimes could be

monitored and a temperature-time history determined, but specimen temperature could not be controlled. Exposure conditions--maximum and minimum temperatures, heating and cooling rates, temperature-time history, and composition of combustion products--were all dependent on production requirements. The disadvantages of this type of materials exposure test are as follows:

- Specimen temperature is not controlled, and sometimes is not measured accurately.
- Flue gas temperature cycles frequently in some furnaces, and therefore specimen temperature also cycles.
- Corrosion data are based on a complicated temperature-time history.
- Corrosion data for a specific temperature are not determined.
- The effect of temperature on corrosion rate is not determined.
- Extrapolation of corrosion data to other furnaces with different temperature-time histories is difficult.
- Stresses induced by thermal cycling cause additional degradation.

Exposure of materials to ambient conditions within a particular furnace might provide useful information for an intended application, especially if the furnace operated normally. Alternatively, the results might serve only as a guide to materials behavior because of atypical temperature-time history. In that case, the results would represent conditions unmatched in other furnaces of the same type, and would not be applicable under a different set of conditions. Furthermore, data interpretation is difficult if temperatures vary over a wide range due to inability to correlate corrosion rates with specific temperatures. As a result, either additional exposure tests would be required or results would have to be estimated. Estimation of corrosion results is unwise if unanticipated failure of a heat exchanger would result in production losses.

A possible solution to this problem is instrumented, temperature-controlled corrosion probes. Ideally, several such probes would be operated at different temperatures of interest in a typical combustion environment. Preselected constant

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temperatures or temperature-time histories would be maintained by heating or cooling as required. In this manner results would be obtained for test conditions representing a particular heat exchanger or for more generic conditions. The main features of a controlled-temperature corrosion probe are as follows:

- Thermocouples for monitoring temperatures
- Internal heating to increase temperature
- Internal cooling to decrease temperature
- Automatic temperature control
- Continuous recording of temperature

Advantages and disadvantages of the two types of exposure tests are presented in Table 1. A disadvantage of the corrosion probe is that relatively few specimens can be tested in the current design, which increases the cost per specimen per test. The conventional exposure test, therefore, could be used to screen a large number of materials, and the corrosion probe could be used to obtain pertinent data for the better materials. Of course, companion specimens having no temperature control could be installed along with the probe(s). Corrosion data representative of the temperature fluctuations of the furnace would be obtained with these specimens. Such data might be useful for a heat exchanger on that particular furnace. The probe data, on the other hand, could be applied to heat exchangers on other furnaces having different temperature-time histories.

The objective of this work was to develop a conceptual design for an instrumented, temperature-controlled corrosion probe suitable for testing candidate heat exchanger materials (both metallic and ceramic) in high-temperature combustion environments. The conceptual design involves size, assembly features, heating and cooling methods, and temperature control instrumentation. The probe has a tubular configuration with temperature control provided by a regulated internal heater and by the flow of cooling air through the bore. The cooling method is similar to that employed in metallic corrosion and fouling probes (Hoffman et al., 1987, Marner and Henslee, 1985, and Marner and MacDavid, 1987), which were designed to

Table 1. Comparison of corrosion test methods

Comparison criteria	Conventional exposure test	Controlled-temperature corrosion probe
Number of materials per test	Many	Few
Expense per specimen	Low	Moderate
Temperature known	Yes	Yes
Temperature controlled	No	Yes
Temperature-time history	Complicated	Simple
Corrosion rate at temperature of interest	No	Yes
Effect of temperature on corrosion rate	No	Yes
Extrapolatable data	No	Yes
Furnace shutdown required for installation or removal	Yes	No

operate at temperatures considerably less than flue gas temperatures by regulating the flow of air through a tubular configuration in contact with the flue gases. None of these probes contained a heat source for increasing the operating temperature.

A computer model was used to evaluate the characteristics of the corrosion probe discussed here. The effects of size, spacings, cooling air flow rate, heater capacity, and flue gas temperature and flow rate were evaluated to determine the possible range of temperature control. The model permitted a theoretical determination of the effect of exposure conditions on probe performance before actual fabrication of the probe.

DESIGN BASIS

Although the probe is intended for use in various furnaces in which the flow rate and temperature of flue gases are different, the heater and air flow were sized for a typical industrial aluminum remelt furnace. The reference furnace burns natural gas with 10% more air than the stoichiometric amount required for combustion. At the highest firing rate (Table 2), the burners produce 4.7 MW (16 E+06 Btu/h) of heat and flue gas temperatures of about 1300°C (2400°F). At the lowest firing rate the quantities are 0.9 MW (3 E+06 Btu/h) and about 750°C (1400°F). The flue gases mostly consist of N₂, CO₂, H₂O, and O₂ as shown in Table 2. Figure 1 illustrates a possible flue gas temperature variation in an industrial furnace. The temperature increases rapidly from 750 to 1300°C, holds for about 2 h, then decreases to 750°C. After 1 h at 750°C, the cycle is repeated. The purpose of the probe control

Table 2. Reference furnace combustion conditions

Combustion products ^a	High fire	Low fire
Heat content		
MW	4.7	0.9
Btu/h	16 E+06	3 E+06
Temperature		
C	1300	750
F	2400	1400
Mass flow rate		
kg/s	1.69	0.29
lb/h	13,385	2295

^a 74% N₂, 13% CO₂, 11% H₂O, 2% O₂.

Figure 1. Example of flue gas vs. corrosion probe temperature.

system is to maintain the probe at a constant temperature during the thermal cycles. Calculations were conducted to determine the amount of internal heating and cooling required to maintain the temperature by adding heat when the flue gas temperature is below the probe setpoint and by cooling when the flue gas temperature is too high.

CHARACTERISTICS OF A CORROSION PROBE

The corrosion probe was designed to provide features that are not available in conventional exposure tests:

- Install in and remove from an industrial furnace flue without affecting furnace operation.
- Automatically maintain the desired temperature within a range of flue gas temperatures and flow rates.
- Provide a continuous, permanent temperature-time history.

The conceptual design is a tubular configuration (Fig. 2) composed of the specimen (test section) and other commercially available components. The probe projects a predetermined distance through a port in the flue wall, and is immersed in flue gases in a crossflow mode. The test section in the reference design is ~2.5 cm OD x 1.9 cm ID x 38 cm long. These dimensions limit the size and capacity of the internal heater. Exposure of the probe may be terminated at any time by withdrawing the probe from the flue wall and closing the port in the wall.

PROBE TEMPERATURE CONTROL

A block diagram of the overall probe configuration and control system is shown in Fig. 3. The thermocouple is mounted along the top (downstream) surface of the probe. Although the bead will be exposed to fouling at this location, it is felt that by being cemented into a recess in the probe at this location a more representative value of average probe temperature will be obtained. Also, the thermocouple will not be exposed to the direct force of the flue gas stream. The thermocouple, which is attached to the outer surface of the test section, provides a signal to the controller. The controller regulates an internal heater and air flow valve to maintain temperature. Actual temperature control is accomplished by a conventional PID controller (e.g., a Barber-Coleman Model 570 process controller). This unit measures probe temperature by means of the thermocouple, and computes an appropriate output signal to drive the control elements, an electric power control, and an air flow control valve. The power

Figure 2. Corrosion probe detail.

Figure 3. Probe assembly/control system block diagram.

controller is a single-phase, 115-V, phase-fired silicon controlled rectifier (SCR) unit. The cooling air flow control is a conventional proportional pneumatic valve, which is driven by an I/P (current-to-pressure) converter. The output from the process controller is a standard 4- to 20-mA signal. The 12-mA point is used as a control "null" point. A signal greater than 12 mA results in the heater being energized, and a signal below 12 mA results in actuation of the cooling air control valve. This configuration provides appropriate control with a minimum of components.

HEAT TRANSFER CALCULATIONS

The steady state effects of probe size, heater output, cooling air flow rate, and flue gas temperature and flow rate on the temperature of the probe were investigated by a computer model. The model calculates the thermal balance in the probe as affected by heat flow due to convection and radiation associated with the flue gas and walls, the heater output, and cooling air flow rate. The calculations were conducted for a SiC corrosion probe having the characteristics shown in Table 3, for flue gases having the characteristics shown in Tables 2 and 4, for a refractory furnace flue having the characteristics shown in Table 5, and for cooling air having the characteristics shown in Table 6. Accurate temperature control requires balancing of the thermal energy received by and transferred from the probe. The balance of thermal energy within the probe (q_s) can be described in terms of the various components:

$$q_s = q_{cg} + q_{rf} + q_{ch} + q_{rh} + q_{ca} \quad (1)$$

where

q_{cg} = convective heat transferred to/from the probe by the flue gases,

q_{rf} = radiative heat transferred to/from the probe by the flue wall,

q_{ch} = convective heat transferred to/from the probe by the heater,

q_{rh} = radiative heat transferred to/from the probe by the heater,

q_{ca} = convective heat transferred to/from the probe by cooling air.

Table 3. Characteristics of SiC probe and heater used in heat transfer calculations

Parameter	Symbol	Value
Probe		
length	L_p	38.10 cm
diameter	D_p	2.54 cm
surface area	A_p	303.8 cm ²
emissivity ^a	ϵ_p	0.9
reflectance ^b	r_p	0.1
wall thickness	t_p	0.32 cm
radius (inner)	r_i	0.95 cm
temperature	T_p	°C (varies with operating conditions)
Heater		
radius (outer)	r_h	0.64
temperature	T_h	°C (varies with operating conditions)
Annulus		
cross-sectional area	A_a	1.56 cm ²
average radius	r_a	0.79 cm

^aStandard Oil Engineered Materials Co., Niagara Falls, New York (formerly Carborundum Resistant Materials Company).

$$r_p = 1 - \epsilon_p.$$

Table 4. Characteristics of flue gases used in heat transfer calculations

Parameter	Symbol	Value
Viscosity	μ_g	0.05 mPa·s
Density		
high fire	ρ_{gh}	0.26 kg/m ³
low fire	ρ_{gl}	0.05 kg/m ³
Thermal conductivity	k_g	0.08 mW/m·K
Mass flow rate		
high fire	$\dot{m}_{gh}$	1.59 kg/s
low fire	$\dot{m}_{gl}$	0.32 kg/s
Velocity		
high fire	v_{gh}	414.1 cm/s
low fire	v_{gl}	82.8 cm/s
Reynolds number	Re_g	528

The convective heat flow between the probe and the flue gas is given by Newton's Law of Cooling:

$$q_{cg} = (T_g - T_p) h_c A_p \quad (2)$$

where

T_g = temperature of the flue gases,

T_p = temperature of the probe,

h_c = convective heat transfer coefficient,

A_p = surface area of the probe.

Table 5. Characteristics of flue used in heat transfer calculations

Parameter	Symbol	Value
Cross-sectional area	A_f	15,000 cm ²
Emissivity ^a	ϵ_{fw}	0.8
Reflectance ^b	r_{fw}	0.2
Geometric view factors ^c	F_{23}	1
	F_{33}	1
	F_{32}	0

^aEstimated.

^b $r_{fw} = 1 - \epsilon_{fw}$ (for solid body).

^c F_{23} = View factor from probe to flue wall.

F_{33} = View factor from flue wall to flue wall.

F_{32} = View factor from flue wall to probe.

Table 6. Characteristics of cooling air used in heat transfer calculations

Parameter	Symbol	Value
Density ^a	ρ_A	0.32 kg/m ³
Thermal conductivity ^a	k_A	0.08 mW/m·K
Viscosity ^a	μ_A	0.05 mPa·s
Specific heat ^a	C_{pA}	1222.6 J/kg·K
Volume flow rate	Q_A	102413 cm ³ /s
Cooling air inlet temperature	T_{IN}	27°F

^aHandbook of Engineering Fundamentals, 3d ed., O. W. Eshbach, ed., John Wiley and Sons, New York, p. 650.

The convective heat transfer coefficient is given by Hilpert (1933):

$$h_c = \frac{k_g \cdot C \cdot Re_g^n}{D_p} \quad (3)$$

where all terms except C and n are identified in Tables 3 and 4. The values of C and n depend on the value of the Reynolds number (Re_g), and were obtained from the following table (Hilpert, 1933).

Re_g	C	n
0.4-4	0.891	0.330
4-40	0.821	0.385
40-4000	0.615	0.466
4000-40,000	0.174	0.618
40,000-400,000	0.0239	0.805

The total radiative heat flow between the probe and the flue wall can be shown to be

$$q_{rf} = \frac{\epsilon_p}{r_p} (R_2 - \sigma \cdot T_p^4) A_p \quad (4)$$

where σ is the Stefan-Boltzman constant (4.805×10^{-13} Btu/ft²·s·R⁴) and new terms except R_2 are identified in Table 3. The term R_2 , the radiosity, is given by

$$R_2 = \frac{\epsilon_p \cdot \sigma \cdot T_p^4 (1 - r_{fw} \cdot F_{33}) + r_p \cdot F_{23} \cdot \epsilon_{fw} \cdot \sigma \cdot T_g^4}{1 - r_{fw} \cdot F_{33} - r_p^2 \cdot F_{23} \cdot F_{32}} \quad (5)$$

where new terms are identified in Tables 3 and 5. The total heat transfer between the probe and its surroundings is obtained by combining the convective and radiative contributions

$$q_t = q_{cg} + q_{rf} \quad (6)$$

Table 2 shows that the flue gas temperature varies between 750 and 1300°C. Either heating or cooling is required to maintain the probe temperature within this range. When the flue gas temperature is below the desired probe temperature, heat is added to the specimen from the internal heater. Initially, electric heating is being employed, although the heat may be provided either electrically or by gas combustion. The electric heater is essentially completely enclosed by the specimen; therefore, in the steady-state condition, the heat input to the probe by convection and radiation is equal to heater input power. Since our heater can provide about 1.6 kW,

$$q_h = q_{ch} + q_{rh} = 1.6 \text{ kW} \quad (7)$$

When the flue gas temperature is above the desired probe temperature, excess heat must be removed from the probe. This is accomplished by flowing a stream of cooling air through the bore of the probe. This component (q_{ca}) of heat transfer can be obtained from the following relation:

$$q_{ca} = \rho A Q A C_p \Delta T$$

$$\left[\frac{h_1 r_h T_h + h_2 r_i T_p}{h_1 r_h + h_2 r_i} \right] \left(1 - e^{-(h_1 r_h + h_2 r_i) \frac{2\pi L_p}{\rho A Q A C_p}} \right) - \rho A Q A C_p \Delta T \quad (8)$$

where

h_1 = convective heat transfer coefficient between the heater and the cooling air as a function of cooling air flow rate,

h_2 = convective heat transfer coefficient between the inner surface of the probe and the cooling air as a function of cooling air flow rate.

The term in brackets in Eq. (8) was developed for design analysis of probe performance as a function of cooling air flow rate, Q_A , heat transfer film coefficients, h_1 and h_2 , and the location within the probe, L_p . The term is equivalent to the actual temperature of the cooling air at the exit of the probe, a quantity that could be measured during probe operation with a suitably placed thermocouple.

Using Eq. (6), the quantity q_t was calculated for the temperature range 750 to 1300°C. Figure 4 shows that q_t , the amount of heat to be added or removed from the probe, increases with increasing difference between T_g and T_p . This figure defines the operating regime of the probe if sufficient heating and cooling capacity are available to maintain the desired probe temperature. Because the available electrical heater can provide only

Figure 4. Controlled temperature corrosion probe operating regime.

1.6 kW of heat, the probe can be maintained at a temperature ~125°C above T_g . Better control can be obtained if cooling is required. The cooling capacity obtained from Eq. (8) allows the probe to operate at a temperature ~375°C below T_g .

SUMMARY

A need to evaluate candidate waste heat recovery materials under essentially constant temperature conditions has been identified. Appropriate modeling and design calculations demonstrate the feasibility of constructing a controlled-temperature corrosion probe. These calculations also indicate that sufficient energy may not be available from existing electric heating elements to control probe temperature. While the initial configuration utilizes such an element, provisions are being made to incorporate a gas burner in future designs to permit a broader operating regime in the heating mode.

A corrosion probe is now being fabricated according to the conceptual design described herein. Fabrication of this probe is intended to reveal assembly problems and to provide a prototype for laboratory testing, which will be conducted in a simulated furnace flue using natural gas combustion products as the flue gases. The responsiveness of the heater and cooling air to various flue gas temperatures will be determined, and control system performance will be evaluated.

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