TOTAL HEMISPHERICAL EMISSIVITY OF REACTOR PRESSURE VESSEL

CANDIDATE MATERIALS: SS 316 L,

SA 508, and A 387 Grade 91

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Doctor of Philosophy

By

FATEN AL ZUBAIDI

Dr. Sudarshan K. Loyalka and Robert V. Thompson. Dissertation Supervisor

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The undersigned, appointed by the dean of the Graduate School, have examined the dissertation entitled:

TOTAL HEMISPHERICAL EMISSIVITY OF REACTOR PRESSURE VESSEL

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presented by Faten Al Zubaidi,

a candidate for the degree of Doctor of Philosophy

and hereby certify that, in their opinion, it is worthy of acceptance.

Professor Sudarshan K. Loyalka

Professor Robert V. Tompson

Professor Zaichun Frank Feng

Professor Ahmed S. El-Gizawy

Professor Ghosh, Tushar

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ABSTRACT

Future nuclear Reactor Pressure Vessels (RPV) require high emissivity materials and alloy for efficient heat decay removal under normal and accidental events. Emissivity is the material property that determines the heat radiation rate from the material surface. The total hemispherical emissivity of candidate materials, 316L stainless steel, A508/A533B steel, and A387 Grade 91 steel were measured in the present work. Standard ASTM C835-06 was used to measure the total hemispherical emissivity of structural materials of RPV. The bell jar emissivity was measured using 304 stainless steel strips coated with black paint (Aremco HiE-Coat 840 M). Testing these strips verified that the internal bell jar emissivity is greater than 0.8 as required by the ASTM standard.

Emissivity of 316L stainless steel was measured for the following conditions: (1) “as-received” from the supplier (un-oxidized) and; (2) oxidized in air at 573 K for up to 3000 h. The emissivity of as-received test strips varied from 0.24 at 434 K to 0.34 at 1026 K. The total hemispherical emissivity of 316 L stainless steel oxidized in air for 500 h or more ranged from 0.28 at 429 K to 0.38 at 1096 K. Increasing the sample oxidation time beyond 500 h at 573 K did not have any significant observable effect on the total hemispherical emissivities of any of the test strips studied. It is suspected that the emissivity ceased to increase during the additional oxidation time because of the protective chromium oxide layer formed on 316L stainless steel inhibiting further oxidation.

The total hemispherical emissivity of A508/A533B steel was measured in the temperature range from 400 K to 900 K for the following surface conditions: (1) mirror polished finish; (2) mirror polished and oxidized in air at 573 K and 773 K for various durations ; (3) after Electric Discharge Machining (EDM) cutting process; and (4) EDM and oxidized in air at 573 K and 773 K up to 1000 h. The emissivity of polished A508/A533B (un-oxidized) strips varied from 0.16 to 0.24 within the temperature range from 552 K to 1180 K. Emissivity values for polished samples oxidized in air for 10 h at 573 K were increased from 0.18 at 543 K to 0.28 at 815 K. While the emissivity of polished samples oxidized in air for 100 h at 573 K increased from 0.16 at 460 K to 0.33 at 815 K. The total hemispherical emissivity of EDM cut and oxidized in air at 573 K for 500 h has experienced an increase from 0.75 to 0.8 in the range of temperature from 418 K to 625 K. No further significant change in emissivity was observed following an increase in oxidation time from 500 h to 1000 h. Polished and oxidized strips at temperature 773 K for 10 h has increased from 0.74 at 464 K to 0.81 at 838 K. This strong effect of temperature has not been noticed for EDM-cut oxidized samples at 773 K for 500 h due to detach of the oxide layer from the A508/A533B substrate.

The emissivity of A387 Grade 91 was measured for the following conditions: (1) EDM cut; (2) EDM cut and oxidized in air at 873 K and 1023 K for up to 5 h; (3) sandblasted with 320 grit size; and (4) sandblasted with 320 grit size and oxidized in air at 1023 K for 5 h. The emissivity of EDM cut A387 Grade 91 varied from 0.57 at 444 K to 0.62 at 1026 K, while the emissivity of A387 Grade 91 sandblasted with 320 grit increased from 0.49 at 464 K to 0.57 at 1048 K. The total hemispherical emissivity of A387 Grade 91 oxidized in air at 873K for 1 h has increased from 0.75 to 0.8 in the

temperature range of 420 K to 831 K. The emissivity of A387 Grade 91 oxidized in air at 1023 K for 1h increased from 0.69 at 429 K to 0.8 at 889 K. Oxidation of A387 Grade 91 in air at 873 K and 1023 K did not result in significant difference in emissivity values among various test strips for oxidation durations of 1 h, 3 h, and 5 h. The emissivity of A387 Grade 91 sandblasted with 320 grit and oxidized at 1023 K for 5 h increased from 0.63 at 440 K to 0.74 at 944 K.

CHAPTER 1 INTRODUCTION

1.1. Background

The fast-growing demand for clean energy to conserve the environment has contributed to increase investigations to develop nuclear energy among other energy sources.¹ Nuclear energy remains an important source of electricity.² Recently, Nuclear power represents 20% of United States electricity demands without carbon emission. The U.S. Department of Energy (DOE) has worked to improve the safety and environmental standards for nuclear power by investigating other possible nuclear fuel technologies for current Light Water Reactors (LWRs).³ Light Water Reactor (LWR) has been the major source of electricity for the past sixty years with a power output increase from 60 MW to 1600 MW.⁴

Major efforts have been made to increase the efficiency of Light Water Reactors (LWRs) and define the safety analysis under nominal and accident conditions. In the US the Pressurized Water Reactors (PWRs) and the Boiling Water Reactor (BWR)⁵ are the two most popular versions of such reactors. The Light Water Reactor Sustainability (LWRS) Program will extend the life expectancy of nuclear power plants beyond the current 60-year licensing period and assure long-term reliability, productivity, safety, and security. Idaho National Laboratory serves as the Technical Integration Office and

manages the Research and Development (R&D) projects in the following areas: Materials Aging and Degradation Assessment, Advanced Instrumentation, Information and Control Systems Technologies, Risk-Informed Safety Margin Characterization, and Advanced Light Water Reactor Nuclear Fuels. Because of nuclear industry has a remarkable financial motivation to expand the life of existing plants, the US department will work to guarantee the activities are cost-shared to the maximum extent possible.⁶

For safety, the pressure vessel is an important part of LWRs.^{7,8} Radiation dominates the decay heat removal from a reactor vessel under accident scenarios where coolant is not available. Since heat loss by radiation is mainly proportional via the emissivity to the equivalent black-body radiation at the same temperature, which depends upon the fourth power of temperature, higher emissivity materials are the most desirable structural materials to use in the event of a reactor accident involving a loss of coolant (LOCA).⁹⁻¹²

1.2. Candidate Materials and Challenges

Innovative generation of nuclear reactors requires further development in reliability, economy, and safety. Material selection plays a significant role in nuclear system designs for next generation.^{13,14} Stainless steel has been used in nuclear reactors for decades and is still the best option for some Generation IV reactor designs. Research and Development (R&D) programs have worked to develop new types of steel in order to add a larger capacity and to increase the lifetime of nuclear power plants. Austenitic steel (like 304&316) is the most often used steel in pipes and other vessel components structures due to its mechanical properties and corrosion resistance at high temperatures. Light Water Reactors and Very High Temperature Reactors tend to use this type of stainless steel.^{15,16}

In current LWRs, the pressure vessels are built of a low ferritic steel, like SA508 class 2 and A533 grade B.¹⁷ The pressure vessel has a large wall thickness of 450 mm in cylindrical part and exposure to feed water at 280 ° C. That avoids thermal stresses and deformation problems.⁴ A508 is being used in China in nuclear power plants as a viable material for pressure vessel reactors. A387 Grade 91 ferritic alloy is considered a candidate material for Very High Temperature Reactor (VHTR) Pressure Vessel (PV) and the power conversion vessel.¹⁸ Alloy 617, Haynes 230, Inconel 800H and Hastelloy X are most likely to be used for the Intermediate Heat Exchanger (IHX) of nuclear reactors. Many of other materials such as SA-213 and SA -335 have undergone extensive testing regarding to high temperature strength, corrosion resistance, etc., but less data has been available pertaining to emissivity.

1.3. Emissivity

In a vacuum environment, radiation is the only significant mode of heat transfer, since radiation energy can transfer without medium between objects. Any material surface can emit and receive radiation energy from other objects surrounding it.¹⁹ A black body is a reference surface, which emits all the radiation energy at given temperature and wavelength. On the other hand, it can absorb a maximum amount of radiation energy incident on its surface.²⁰

The radiation energy emitted from a material's surface with respect to radiation energy emitted from a black body at the same temperature is called ***Emissivity***.¹² Emissivity plays a significant role in thermal measurements and radiation heat analysis for scientific and engineering applications. Emissivity is a surface property –it is affected by surface conditions like surface roughness and oxidation.²¹ In addition, it depends on surface temperature, emission direction and emission wave length. Emissivity can be total emissivity (over all wavelengths), hemispherical emissivity (all angles) or total hemispherical emissivity (over all wavelengths and angles). For Reactor Pressure Vessel (RPV), a material with high surface emissivity is important for decay heat removal under accident conditions due to passive cooling of the RPV by radiation from the outer surface to the air in the cavity between the RPV and surrounding concrete.

1.4. Objective of this study

The objective of this work was to acquire and report data on total hemispherical emissivity according to standard method (ASTM C835- 06)²² of Reactor Pressure Vessel candidate materials as a function of temperature under long and short- term oxidation in air. The data were obtained with a measurement system built in compliance with all of the requirements of ASTM C835-06²² and following the data acquisition procedure described therein. We also estimated the uncertainty in the data using techniques described elsewhere.²³(Kyle's paper) The surface oxidation of the strips was measured with a Scanning Electron Microscope (SEM) and surface roughness was analyzed using optical profilometry.

1.5 Dissertation Organization

This dissertation is organized as follows:

Chapter I provide a short background on LWRs and a summary of the objectives of this work and the organization of this dissertation.

Chapter II presents the literature review of the previous work on emissivity measurements for the three candidate materials.

Chapter III describe the experimental procedure including experimental method, apparatus, and oxidation process.

Chapters IV, V, and VI compose the main parts of this dissertation. Each chapter provides the total hemispherical emissivity measurements of candidate materials SS316 L, A508/A533B, and A387 Grade 91. These chapters include material and sample preparation, experimental results, discussions, and conclusion for each candidate material, separately.

CHAPTER 2 LITERATURE REVIEW

Emissivity is a physical property which is defined as the ratio of radiative energy released from material to that of a blackbody at the same temperature that is $\epsilon = \frac{S_{sample}}{S_{blackbody}}$. Because emissivity is a surface phenomenon, chemical composition, roughness, porosity, and oxidation of the material surface, affect it. This chapter provides below a review of some significant research to date for emissivity measurements and the various parameters that have remarkable influence on the measurements.

2.1. Total hemispherical emissivity measurements using standard

ASTM C835-06

Numerous studies have been conducted to measure total hemispherical emissivity for different materials using standard ASTM C835-06.²² Maynard et al. (2010,2012)^{24,25} published a series of reports to measure the effect of surface roughness, oxidation, and graphite coating for two different Nickel based alloys, Hastelloy X and Hayne 230. Four sets of samples were used; “as received”, roughening with various grit size (100, 240, 600) sand paper, oxidized in air at temperatures 1153 K and 1163 K for 5,10, and 15 min and coated with graphite thin layer and various coating thicknesses. The emissivity increased with finer grit size used; however, the graphite layer showed significant increases in

emissivity values. The authors noticed that an increase in graphite layer thickness increases emissivity, which is still lower than the pure graphite emissivity, this means more investigations are needed to specify the optimal layer thickness.

Gordon et al. (2012)²⁶ measured the total hemispherical emissivity of Hastelloy N using standard ASTM C835 -06 method in the temperature range of 473 K to 1498 K. Based on their measurements, emissivity changed from 0.22 to 0.28 in the temperature range of 473 K to 1498 K for structural Hastelloy N. A small increase in emissivity was observed in oxidized samples. While a remarkable increase in emissivity values was observed in graphite-coated samples. It can be noticed from literature, Hastelloy X and Hastelloy N have close values of emissivity even both contain different amount of Nickel. This indicates that both materials have the same behavior.

The emissivity of Inconel 718, the candidate material of Very High Temperature Gas Reactors (VHTGRs) was measured by (Keller et al., 2015)²⁷ for various surface conditions; 1) oxidized in air and humidified helium, 2) roughening surface by aluminum oxide powder sand blasting, and 3) coated with graphite powder, under temperature interval from 600 to 1250 K. No significant effect of oxidation in air and humidified helium was observed. However, the emissivity values were doubled when a surface sandblasted with aluminum oxide at grit size 220 via as received surfaces. It is clearly noticed that graphite powder coated on 220-grit surface increases emissivity in range from 0.6 to 0.7 at range of temperatures from 650 K to 1200 K.

2.2. Materials Emissivity

2.2.1. Emissivity of SS 316L Alloy

Isetti et al. (1979)²⁸ investigated the effect of surface roughness and oxidation on total normal emittance of 316L stainless steel using optical systems. Oxidation was obtained at temperature up to 1073 K for 180 h in air environment. The results of this research showed that the behavior of polished and rolled (as-received) samples was the same, emittance increased as temperature increased, however the values were two or three times higher for high surface roughness which ascribed to the thick oxidized layer of roughed samples.

Isetti and Nannei (1980)²⁹ reported the significant influence of surface finish on total normal emittance of SS316L oxidized samples. Two different sets of samples were polished (metallographic & mechanical polishing) with the same average roughness and oxidized in air at temperature 1100 K for 3 h and 14 h. The results showed that the emittance values increase was about 2.5 greater when the surface was polished metallographically as opposed to mechanical polishing due to different polishing method. A further study by Isetti (1980)³⁰ measured emissivity for oxidized SS316L in air at temperature of 1050 K for up to 48 h. The emissivity values increased sharply within the first 30 minutes and then decreased between 30 and 60 minutes. In general, the results showed that increasing oxidation time leads to increase emissivity.

Bartolini et al. (1985)³¹ observed that the emissivity of SS316L increased with oxidation time in both annealed and un-annealed samples, which were both, oxidized in air at temperature 900 K for up to 25 h. The total normal emittance values of un-annealed

samples were greater than the emittance values of annealed samples. This difference in emissivity values is ascribed to the thicker oxide layer formed on annealed sample surfaces. At the same time, the authors studied the effect of electro-polishing process on their samples after chemical etching. This treatment eliminates the difference in emissivity values between the two types of initial polishing processes were used.

Frachet et al. (1986)³² measured the total hemispherical emissivity of SS316L for both increasing and decreasing temperatures using a calorimetric method, a vacuum of 10^{-5} mbar. The chamber was water cooled with a known emissivity. The results indicated that the total hemispherical emissivity increased with increasing temperature.

Groot and Dvorak (1991)³³ published the emissivity measurements of two different coating methods (atmospheric and vacuum plasma spray) to deposit titanium carbide (TiC) on SS316L surface. To investigate the effect of plasma spray technique, various powder grain sizes were applied. Emissivity measurements obtained at a temperature of 525 K and in a range of temperatures from 700 K to 1150 K. As a result, emissivity increased with the presence of oxygen. Further study on the effect of TiC layer thickness is needed.

2.2.2. Emissivity of A508/A533B and A387 Grade 91

A limited number of studies have reported on the spectral emissivity of A508/A533B and A387 Grade 91 (T91). Cao et al. (2012)³⁴ measured the spectral emissivity of polished samples SA508 & SS316 in situ air oxidation at temperature 500 °C for 2-4 h and at temperature 700 °C for 1-5 h. The behavior of both materials was the same, spectral emissivity rose when the temperature, time, and wavelength increased. Because of stainless steel 316L has a high level of oxidation resistance due to a high amount of chromium in this steel, it has lower emissivity values than SA508. The difference in emissivity values for both materials when temperatures increase results from the development of an oxide layer on the surface.

In additional data, Cao and colleagues (2013)¹⁰ measured the emissivity of SS316L and SA508 at high temperature in air. Samples were roughened using Carbide Silicon (SiC) paper with different grit sizes and polished with 0.5µm silica colloidal solution. Samples were then oxidized at temperatures 500°C and 700°C for up to 5 hours. SS316L has lower emissivity values than SA508 due to the high content of chromium in SS316L that builds a protective layer inhibiting further oxidation. However, no significant change in the emissivity values of SA508 was observed with oxidation time.

Azmeh et al. (2016)³⁵ obtained the total hemispherical emissivity of A387 Grade 91 at temperature range from 325 K to 1299 K for different surface circumstances. Electrical Discharge Machining (EDM) technique was used to cut samples and then sandblasted with a mild abrasive to clean the surface. Their results showed that the emissivity for mildly abraded samples increased via temperature increase and decrease at

temperatures up to 1100 K. The authors observed the same performance for oxidized and sandblasted samples. The reason behind this is not known. The emissivity of graphite-coated surfaces was less than the emissivity of pure graphite, which can be ascribed to a thin graphite layer coated on the surface.

Jo et al. (2017)³⁶ measured the spectral directional emissivity for oxidized SA508 for various temperatures and durations, and roughened samples. Emissivity measurements were taken at room temperature for a range of the wavelengths. Experimental results showed that both oxidation and surface roughness affect spectral emissivity with oxidation having a significant effect. Emissivity increased with oxidation temperature and time at shorter wavelengths.

Recently, King et al. (2016,2017)^{37,38} reported data on spectral directional emissivity for SA508 and A387 Grade 91 under several surface conditions. They observed heavy oxidation of SA508 at high temperature. Their data is deemed a reference for excessively oxidized materials. The results have a good agreement to the work of Cao et al.¹⁰ While A 387 Grade 91 steel with high content of chromium (Cr) developed a thin oxide layer resulting in lower emissivity values. The oxidation effect was greater than the roughness on the emissivity values.

2.3 Summary

- ❖ Some of researchers used ASTM standard C835-06 method for various materials. (Hastelloy X, Hastelloy N, and Inconel 617, Inconel 718).
- ❖ Most of work on SS316 was to measure total normal emissivity. One paper measured total hemispherical emissivity by Frachet et al. (1986).
- ❖ Few works measured spectral emissivity of SA508/A533 B and A387 Grade 91.
- ❖ Azmeh et al. (2016) measured the total hemispherical emissivity of A387 Grade 91.

The goal of this study is to measure the total hemispherical emissivity of three candidate materials for Reactor Pressure Vessel and can be used as basis for further research.

CHAPTER 3 EXPERIMENTAL PROCEDURE

3.1. Experimental Method

ASTM Standard C835-06²² is a calorimetric test method based on vacuum environment, it used to measure total hemispherical emittance of metals, graphite surface, and coated metal surfaces at temperature up to 1673 K. This method is used to determine the effect of oxidation on emittance. The total hemispherical emissivity measurements based on ASTM C835-06 can be determined with the equation:

$$\varepsilon_1 = \frac{Q}{\sigma A_1 (T_1^4 - T_2^4)} \quad (1)$$

Where:

$Q = IV$ is the power radiated from the surface of a given test section on a sample strip.

Where I is the current applied and V is the voltage.

A_1 is the surface area of the test section of sample strip.

T_1 and T_2 are the temperatures of the test section and of the vacuum chamber wall respectively.

σ is the Stefan- Boltzmann constant.

The samples of material are placed in evacuated chamber and put between two direct-current electrodes (dc). The direct current (dc) heats the sample strip which is approximately 13 mm wide and 250 mm long. ASTM gives some requirements that reduce the systematic errors. To apply this method, the chamber dimensions must satisfy the following condition:

$$\varepsilon \gg \frac{A_1}{A_2} \left(\frac{1}{\varepsilon_2} - 1 \right) \quad (2)$$

Where:

ε_2 : total hemispherical emissivity of the chamber wall.

A_1 : surface area of the test strip over which heat generation is measured (cm²), as in equation (1)

A_2 : total internal surface area of the chamber (cm²).

ASTM requires $\frac{A_1}{A_2} < 0.01$ and chamber wall emissivity is greater than 0.8.

3.2 Experimental Apparatus

The apparatus is shown in Figure 3.1 was used to measure the total hemispherical emissivity of corroded sample stripes. It was constructed according to standard ASMT C835-06 protocol. Mainly, it consists of a large bell jar on the base wall, vacuum pumping station (mechanical pump, molecular turbo pump), two data acquisition unit (voltage, temperature), pressure indicator, power supply, and sample holder which is made of stainless steel, works as electrical conductor and is deemed a part of the heating system.

The sample holder is fixed at the top end and is used as the mount for another solid steel arm which can move upward and downward to hold sample stripes of varied lengths.

Prior to experiment runs, the inside of bell jar black paint was removed by sandblasting it using silicon carbide (SiC) blasting sponge with 60 grit size. Then, paint the bell jar and the disk inside the base wall with two coats of black paint (Aremco HiE- Coat 840 M) having an emissivity greater than 0.9. The base wall has an aperture in the bottom center to connect the turbo pump to the system. In addition, there are eight ports on the base wall to feed through. The bell jar dimensions are 45.7 cm in diameter and 59.7cm in length with total internal surface area about 11. 850 cm². To degas the system before measurements, heating Tape is wrapped around the bell jar.

A sample stripe is in dimensions (25.4 cm long, 1.27 cm wide and 0.0254 cm thick). The sample stripe of the material being studied is suspended at the center of the bell jar using a - sample holder. The test section used in this work is the region between two ASTM test sections as shown in Figure 3.1 to ensure the emissivity calculations refer to the center of the sample strip. Dead weight is about 0.5 g is suspended from the lower end of the sample strip to avoid accidentally flexing during thermal expansion of the sample strip.

The vacuum pumping system consists of a turbo-pump and a mechanical roughing pump to maintain a vacuum of 10^{-6} to 10^{-7} mmHg. The system pressure is measured during experiments using cold cathode pressure gauge (CC gauge) pressure readout. Power supply is used to provide the system power and is connected to the sample strip. Flux-gate magnetometer sensor was used for even more accurate measurements to measure the

current flow. Note thermocouple feedthroughs, pressure gauges, and data acquisition are shown in Figure 3.2. Type K thermocouple wires are attached to sample stripes using spot welding and connect to thermocouple feed through by thermocouple connector which is mounted on ceramics thermocouple connectors. A flexible cable is used to connect the lower electrical end with the electrical feed through.

(a) Emissivity Apparatus

(b) Sample

Fig. 3.1. (a) An illustration of the main elements of the emissivity measurement setup and electrical circuit for specimen heating: (1a) vacuum chamber base well, (1b) vacuum chamber bell jar (high emissivity coating on inner wall); (2) ceramic thermocouple mounting posts for easy connection of specimen thermocouples to thermocouple feedthroughs; (3) sample strip; (4) dead weight to prevent sample strip from buckling; (5) current shunt resistor; (6) DC power supply; (7) polarity reversing double-pole double-throw switch; (8) hall effect sensor, (9) mechanical roughing pump; and (10) turbomolecular pump. Note thermocouple feedthroughs, system vent, pressure gauges, and data acquisition are not shown. (b) An illustration of the test section at the center of the strip. Five spot welded Type K TCs are spaced 1.27 cm apart with the voltage drop measurement ΔV_1 - ΔV_4 , across each 1.27 cm section.

Figure 3.2 Photo of the emissivity showing the vacuum station, data acquisition, power supply and pressure gauges.

3.3 Oxidation

3.3.1 Long-term oxidation

The long-term oxidations were obtained using HZS 12/600 three zone tube furnace (Carbolite Gero Ltd) as shown in Figure 3.3- a to ensure a uniform oxidation layer for all strip lengths and to treat more than one sample strip at the same time. The furnace has a uniformity of $\pm 5^{\circ}\text{C}$ over a length of 500 mm and has 8 segments. Each segment is held at given temperature for 100 h. The manufacturer's setting for heating rate is $5^{\circ}\text{C min}^{-1}$ to minimize any thermal shock that might result in process tube breakage. The air flow rate used during oxidation was 150 mL min^{-1} . Eight strips were placed in the furnace at the same initial time allowing for the simultaneous oxidation of different strips for different time periods under essentially identical conditions. The strips were exposed to air in the furnace at a constant temperature of 573 K for up to 3000 h. All of the sample strips were weighed individually both before and after oxidation, but no detectable weight change was observed in any of them.

3.3.2 Short-term oxidation

The short-term oxidations were performed using the same HZS 12/600 three zone tube furnace (Carbolite Gero Ltd) illustrated above and the same procedure. The strips were exposed to air in the furnace at high temperatures (825 K and 1023 K) for 1 h, 3 h, and 5 h. To assure there is no oxidation occurring during heating and cooling time for the furnace to reach the exact temperature, we added vacuum pump to the furnace setup as shown in Figure 3.3-b. The vacuum pressure that was used is between 100 – 300 mtorr. At this pressure, the furnace heating program was started. When the furnace reaches the desired

temperature, the vacuum pump is turned off and the air flow goes into the furnace to start oxidation. Then, the oxidation time is complete, the vacuum pump is turned on again until the furnace cooled off. Figure 3.2 shows the experimental set up of long-term and short-term oxidation of candidate materials (SS316L, A508/A533B, and A387 Grade 91).

3.4 Experimental Procedure

Once the oxidation process is complete, five thermocouples are attached to the strips. The strips are washed from any contamination using a bath of acetone followed by a rinse of deionized water. The strip is secured within the sample holder, and each thermocouple is attached to its corresponding feed-through. According to ASTM C835-06, the chamber is evacuated to an initial pressure of 5×10^{-2} torr. When the pressure is obtained, the heat is initiated using heating tape to help degassin the system. Once the pressure reaches 1×10^{-5} torr, the degassing is complete. The test strip is resistively heated when the internal chamber temperature is 295 K.

(a)

(b)

Figure 3.3 Illustrate the three-zone furnace (a) for long-term and (b) for short-term oxidation.

CHAPTER 4 EMISSIVITY OF 316L STAINLESS STEEL

4.1 Introduction

Data on the radiative properties of various kinds of stainless steel are required for estimations of heat transfer in nuclear systems (pipes, vessels, and other component structures).³⁹ Radiation dominates the decay heat removal from a reactor vessel under accident conditions where coolant is not available. Since heat loss by radiation is mainly proportional via the emissivity to the equivalent black-body radiation at the same temperature, which depends upon the fourth power of temperature, higher emissivity materials are the most desirable structural materials to use in the event of a reactor accident involving a loss of coolant (LOCA).^{9,10,11}

Emissivity is fundamentally a surface property of a material and is defined as the ratio of radiated energy emitted from a material's surface to the radiated energy that would be emitted from a black body at the same temperature. Emissivity can be strongly affected by a number of surface conditions such as oxidation, coating, roughness, etc.^{26, 36, 40-43} There have been a number of studies demonstrating the effect of oxidation on the total hemispherical emissivity of various materials.^{25, 35, 44, 45}

The emissivity of SS316L for different surface conditions has been measured by several authors. The literature on emissivity measurements of SS316L has reviewed in chapter two. Hunnewell *et al.*¹² measured the effect of roughness, coating, oxidation and a combination of coating or oxidation with roughness on the total hemispherical emissivity of SS316L. The strips were oxidized at 973 K for 5, 10, and 15 minutes. As expected,

emissivity increased overall with oxidation, but anomalous results were observed between the 10 and 15 min oxidation samples.

The effect of surface conditions (roughness and oxidation) on the total hemispherical emissivity of two iron-based alloys (these were identified only as samples A and B, and the actual compositions were not given) has been investigated by Fu and colleagues.²¹ A steady-state calorimetric method was used in their work and the results showed that the total hemispherical emissivity increased with surface roughness and that this effect became gradually smaller at elevated temperatures. They also observed that the emissivity values of oxidized samples at 673 K were similar to each other for a variety of oxidation times. Cao *et al.*^{10,34} measured the effect of oxide layer thickness on emissivity at temperatures of 773 K and 973 K for various time periods. Their finding showed that the temperature has a stronger effect on the oxidation than the oxidation time. This means that the oxide layer will become thicker at higher temperatures which increases the emissivity but that the emissivity will be relatively insensitive to how long the sample remains at the higher temperature. Also implied is that the maximum increase in the emissivity of an oxidized sample occurs at the maximum temperature that it experiences and that subsequent decreases in temperature do not necessarily imply any decrease in the changed emissivity.

The objective of this chapter is to acquire and report data on total hemispherical emissivity of SS316L over the range of temperatures from 400 K to 1100 K following long-term sample oxidation of up to 3000 h in air at a temperature of 573 K. The data were obtained with a measurement system built in compliance with all of the requirements of

ASTM C835-06²² and following the data acquisition procedure described therein. We also estimated the uncertainty in the data using techniques described elsewhere²³.

4.2 Material and Sample Preparation

Large quantities of stainless steel have been used in the nuclear industry due to its excellent mechanical properties and high corrosion resistance at an elevated temperature. Austenitic steels (316) are the most widely used in hot environments where the corrosion is standard. The variety of austenitic steel SS316 comes from the amount of carbon. It can be low carbon austenitic steel (316L) or high carbon austenitic steel (316H). Light Water Reactors and Very High Temperature Reactors tend to use austenitic steel 316L. The low carbon content in this steel can reduce the grain boundary chromium carbide precipitation which can end up with an intergranular corrosion in many service environments. Austenitic steel (like 304 and 316) is the most often used steel in pipes and other vessel components structures.

The samples were constructed from stainless steel sheets of SS316L that were 30.48 cm long, 25.4 cm wide and 0.0635 cm thick. First, the sheets were cut into strips 1.27 cm wide using programmable water jet cutting technique with a 0.2 mm tolerance. Second, the length of the strip was cut to 25.4 cm using a simple shearing machine. We cleaned these using procedures similar to those of Hunnewell *et al.*¹² The general composition of SS316L and some related properties relevant to present work are given in Table 4.1.

Table 4.1 *Composition and selected properties of austenitic SS316L.*

Grade:	316L
UNS Number:	S31603
Composition (%):	
C	0.030
N	0.10
Si	0.75
P	0.045
S	0.030
Cr	16.0–18.0
Ni	10.0–14.0
Mo	2.00–3.00
Mn	2.00
Fe	Balance
Density (kg m⁻³):	8000
Tensile Strength (MPa):	485
Yield Strength (MPa):	170

We also coated and used some sample strips of 304 stainless steel (with identical dimensions as those of SS316L steel) with black paint (Aremco HiE-coat 840 M) to determine emissivity of the inside surface of the bell jar used for measurements and to ensure compliance with the standards (discussed in chapter 3). The strips were exposed to air in the furnace at a constant temperature of 573 K for up to 3000 h (see long-term oxidation chapter 3). All of the sample strips were weighed individually both before and after oxidation, but no detectable weight change was observed in any of them. We used a Denver balance A-200 DS on scale setting 200 ± 10^{-4} g with maximum capacity of 200 g

and readability of 0.1 g. Prior to oxidation, all strips were cleaned using acetone bath and left them to dry in air.

Surfaces and cross-sections for as-received and oxidized samples were examined with a scanning electron microscope (SEM, FEI Quanta 600). For post oxidation SEM examination, small square samples 1.27 cm long were cut from each strip using a water jet. While it is possible that the emissivity testing itself might have resulted in some alteration of the sample surfaces, this is not very likely as the temperature range studied was not sufficiently high so as to plasticize (partially melt or permanently deform) the samples and the measurements of emissivity are made in a high vacuum pressure between 10^{-7} to 10^{-5} torr so that no additional oxidation should have occurred.

To measure the surface temperature of the strip, five Type K thermocouples were spot welded to the strip. The thermocouples were evenly spaced (1.27cm apart) on the samples and the diameter of thermocouple wires was less than 0.13 mm to minimize and thermal conduction away from the samples. Also, we cleaned the strips as mentioned above before experimental runs.

4.3 Total Hemispherical Emissivity Measurements for SS316L:

Results and Discussion

Since SS304 is the material from which the bell jar in the total hemispherical emissivity measurement system is constructed and since the interior of the measurement system is almost entirely coated internally with the high emissivity Aremco paint, data were first obtained for 304 stainless steel strips (SS304) coated with this same paint. Testing of these strips verified that the internal bell jar emissivity value was greater than 0.8 required by the ASTM standard as shown in Figure 4.1.

The total hemispherical emissivity of as-received SS316L as a function of temperature is shown in Figure 4.2. The emissivity data from Hunnewell *et al.*¹² are also included. The total hemispherical emissivity values in the current work increased from 0.24 at 435 K to 0.34 at 1027 K. The total hemispherical emissivity values obtained in present work also agree with the data reported by Hunnewell *et al.*¹²

Figure 4.1 Total hemispherical emissivity data for 840-M Hi-E coating for as-received substrate.

Figure 4.2 Total hemispherical emissivity of as-received SS316L

For studying the effect of long-term oxidation, six strips were oxidized in air at 573 K. Sample strips were oxidized starting from 500 h up to 3000 h. The emissivity values are shown in Figures 4.3 and 4.4 and numerical values are given in Table 4.2. The emissivity increased by a small amount for all temperatures when the surface was oxidized in air at 573 K for 500 h. No significant increase was observed when the oxidation time at this temperature was increased to up to 3000 h. This is believed to be due mainly to a protective oxide layer formed on the metal surface which we speculate is chromium based. The emissivity values for oxidized strips at 573 K for 1500 h were lower than the emissivity values for oxidized samples for 500 h, which is surprising because the emissivity is expected to increase with increasing oxidation time even if slightly.

Table 4.2 Total Hemispherical Emissivity of SS316L as-received and oxidized in air up to 3000 h

Treatment	Temperature(K)	Emissivity
As-received	434 - 1026	0.25 - 0.34
500 h	429 - 1096	0.28 - 0.39
1000 h	428 - 1034	0.27 - 0.39
1500 h	436 - 1123	0.26 - 0.39
2000 h	435 - 1094	0.25 - 0.38
2500 h	425 - 1089	0.27 - 0.38
3000 h	426 - 1090	0.27 - 0.396

Figure 4.3. Total hemispherical emissivity for SS316L as-received & oxidized in air at 573 K for 500 h,1000 h, and1500 h.

Figure 4.4 Total hemispherical emissivity for SS316L as-received & oxidized in air at 573 K for 2000 h, 2500 h, and 3000 h.

ASTM 835-06²² stipulates that for the measurements to be valid, the retested emissivity value at a certain temperature should change by no more than 2% of the original measured emissivity. To ensure the surface properties have not changed during testing in the same run, we retested measurements (same sample) for some temperatures to determine the temperature limit for changing surface properties. Such upper limit temperatures for surface change of each strip condition are shown in Table 4.3. Figure 7 shows an example for the re-testing temperatures of a sample strip that was oxidized in air at 573 K for 1000 h. The emissivity value changed by 0.72% at 1032 K, 1.49% at 1056 K, 1.45% at 1072 K, 1.89% at 1098 K, and 2.26 % at 1112 K.

Table 4.3 *The temperature limit of changing surface properties for as received and oxidized strips in air at 573 K for various times.*

Oxidation Treatment	Upper limit Temperature (K)
None - As received	1056
In air for 500 h at 573 K	1121
In air for 1000 h at 573 K	1112
In air for 1500 h at 573 K	1141
In air for 2000 h at 573 K	1128
In air for 2500 h at 573 K	1128
In air for 3000 h at 573 K	1106

Figure 4.5 Emissivity data for SS316L oxidized in air at 573 K for 1000 h and re-testing temperatures, percentage change following re-testing is shown below the re-test data.

For all emissivity measurements, voltage across the sample was read with an analog voltage DAQ card, and LEM HTA-500S sensor was used to measure electrical current flow except for oxidized strips in air at 573 K for 1500 h and for 3000 h. For these two latter strips, a LEM IT 205-S sensor with greater accuracy became available and was used instead. The difference between the two sensors can be seen in Figure 4.6.

Fig. 4.6. Total hemispherical emissivity for SS316L oxidized in air at 573 K for 1500 h showing the close agreement between the two sensors.

Energy dispersive X-ray spectra (EDS) of unoxidized and oxidized surfaces were obtained at three separate locations on a single test section. The averaged values of the elemental composition for the three locations are shown in Table 4.4. It appears that the oxygen content first increases and then gets saturated because of a chromium oxide layer. Figure 4.7-a shows the backscattered electron (BSE) SEM cross-section micrograph of as-received strip. Figures. 4.7-b, 4.7-c, and 4.7-d show the backscattered electron (BSE) SEM cross-section micrographs of three different samples oxidized for 500 h, 1500 h, and 3000 h, respectively.

Table 4.4 *Semiquantitative analysis of the energy dispersive X-Ray spectra of Oxygen and selected constituents of SS316L on the surface for as-received and SS316L as-oxidized for various time periods. The values reported are the average of three measurements.*

Treatment	EDS Normalized concentration (wt.%)				
	O	Fe	Cr	Ni	Mo
As-received	0.53 ± 0.06	69.07 ± 0.21	15.49 ± 0.08	9.72 ± 0.12	1.02 ± 0.01
500 h	2.12 ± 0.19	68.71 ± 1.36	15.41 ± 0.22	9.55 ± 0.26	2.04 ± 0.76
1000 h	2.45 ± 0.03	68.71 ± 0.06	15.35 ± 0.09	9.45 ± 0.04	1.98 ± 0.03
1500 h	2.41 ± 0.04	68.29 ± 0.31	15.43 ± 0.04	9.37 ± 0.12	1.86 ± 0.04
2000 h	2.36 ± 0.06	68.33 ± 0.08	15.36 ± 0.07	9.41 ± 0.08	2.01 ± 0.02
2500 h	2.76 ± 0.07	67.97 ± 0.06	15.51 ± 0.05	9.34 ± 0.08	1.86 ± 0.02
3000 h	2.30 ± 0.67	68.62 ± 1.21	15.52 ± 0.45	9.19 ± 0.33	2.05 ± 0.15

Figure 4.7. SEM micrographs (BSE) showing cross-section of SS316L (a) as-received, (b) 500 h, (c) 1500 h, (d) 3000 h.

The BSE signal shows that a very thin oxide layer formed at some point during the long-time oxidations but exactly when this occurred was not determined. Increasing the oxidation time apparently increases the total hemispherical emissivity by a small amount. However, this increase is not remarkable even for the longest oxidation times considered. For instance, when the temperature of the sample being measured is raised to 1100 K, the relative difference of total hemispherical emissivities for 500 h and 3000 h oxidation is only 2.3%. This implies that the effect of oxidation time on total hemispherical emissivity is minimal at least at 573 K. The effect of oxidation times on samples being oxidized and lower temperatures was not studied although presumably there exists some lower temperature regime where oxidation times on the order of what were studied here might be expected to have a noticeable effect on the emissivity of the material. Certainly, oxidation times at room temperature can be expected to be significantly longer than any of the oxidation times studied here for 573 K. This means the oxide layer grew rapidly at initial oxidation, and this oxide layer then limited further availability of oxygen to the SS316L substrate. Apparently, chromium migrates to the surface, and forms a protective chromium oxide layer.

Oxidation temperature rather than time appears to have the strongest effect on oxide layer growth as Hunnewell *et al.*¹² reported. They reported that the total hemispherical emissivity of SS316L oxidized at 973 K increased with increasing oxidation time. The reason may be that the oxide layer grown at high temperature is generally compact and thin and, thus, at higher temperatures, the long-term, time dependent transport (diffusion) of additional oxygen through this layer to the metal substrate was enhanced by the higher temperature and was thus able to thicken the initial oxide layer that was formed. Since

surface oxide layers that form on metals generally have higher emissivities than their base metals due to the dielectric characteristics of the metal-oxides the emissivity will be higher as the oxide layer thickens to the point that only the emissions of the oxidized surface can be detected.⁴⁶ For optically thin oxide layers, the base metals will be able to contribute to the emissions and this makes the overall emissivity a function of the emissivities of both the oxide and the base metal.

4.4 Conclusion

Total hemispherical emissivity of SS316L has been measured in the temperature range of 430 K to 1100 K. The sample strips had been oxidized in air at 573 K over the times 500 h, 1000 h, 1500 h, 2000 h, 2500 h, and 3000 h. The total hemispherical emissivity of as-received SS316L ranges from 0.25 at 430 K to 0.34 at 1026 K, the emissivity of SS316L oxidized at 573 K for 500 h or more ranged from 0.28 at 429 K to 0.39 at 1096 K which is generally about a 12-15 % increase in emissivity over the entire temperature range studied. This can readily be attributed to the oxide layer initially formed sometime during the first 500 h of sample oxidation. Increasing the sample oxidation time beyond 500 h at this temperature did not have any significant observable effect on the total hemispherical emissivities of any of the samples studied indicating that a protective oxide layer forms earlier than 500 h at 573 K and is subsequently essentially impervious (at 573 K) to the ingress of additional oxygen that might thicken the oxide layer and further increase the total hemispherical emissivity. Taking the current results into account in conjunction with the previous results of Hunnewell *et al.*¹² clearly suggests that it is the oxidation temperature rather than the oxidation time that is the critical factor governing the oxide layer growth and hence the change in the emissivity of the material. Thus, one might expect that during a reactor accident scenario involving air ingress into a reactor using SS316L the radiative properties of the structure will depend most heavily on the maximum temperature reached in the presence of the air rather than how long the structure is actually exposed.

CHAPTER 5 EMISSIVITY OF A508/A533 B STEEL

5.1 Introduction

To achieve high thermal efficiencies, some of the new concepts for next generation light water nuclear reactors are directed at increasing the operating temperatures.⁴⁷⁻⁴⁹ For safety, an important part of LWRs is the pressure vessel.^{7,8} Radiation dominates the decay heat removal from a reactor vessel under accident conditions where coolant is not available. Higher emissivity materials are the most desirable structural materials to use in the event of a reactor accident involving a loss of coolant (LOCA).⁹⁻¹²

Due to their high strength and moderate radiation resistance^{50,51}, A508/A533B low alloy steels have been of interest in developing reactor pressure vessels to increase the power capacity and operational life of nuclear systems. A limited number of studies have been reported on spectral emissivity of A508/A533B. Cao et al.^{10,34} measured the normal spectral emissivity of A508 oxidized in air at temperatures of 773 K and 973 K up to 5 h. They investigated the effect of oxide layer thickness on emissivity for various spectral lengths. Jo et al.³⁶ also studied the spectral normal emissivity of A508, roughened as well as oxidized at various temperatures and up to 200 h. Experimental results showed that both surface oxidation and surface roughness affect spectral emissivity, with surface oxidation having a strong effect particularly at shorter wavelengths. Their experimental results agreed well with those from a simulation model. Spectral directional emissivity of A508 was measured by King et al.³⁸ under various oxidation conditions. Significant increases in emissivity were observed due to oxidation. In general, thin oxide films formed on metals

have higher emissivity than the metals due to the dielectric characteristics of the metal-oxides.⁴⁶

We report here on the measurements of total hemispherical emissivity (using standard ASTM C835-06²² calorimetric test method) of A508/A533B low alloy steel over the range of temperatures from 400 K to 900 K following long-term oxidation up to 1000 h in air at temperatures of 573 K and 773 K. The emissivity of polished and electric discharge machining EDM-cut (un-oxidized) A508/A533B was also measured within the temperature range from 552 K to 1180 K. Test samples (strips) were cut using wire electrical discharge machining (EDM). Surfaces treatments investigated included, 1) polished, 2) EDM-cut, 3) polished and oxidized, and 4) EDM-cut and oxidized. Oxidations were carried out in air up to 1000 h and at 573 K and 773 K. The surface oxidation of the strips was measured with a scanning electron microscope (SEM) and surface roughness of polished strips and EDM cut strips was analyzed using optical profilometry.

5.2 Material and Sample Preparation

The A508/A533B test material was provided to us by Idaho National Laboratory. The material was dual certified as it was forged then hot-rolled into a plate. A508/A533B is a candidate material for reactor pressure vessel that operates in temperature up to 644 K according to ASME code specification.⁵² Strips were cut using wire electric discharge machining (EDM) to the following dimensions $25.4\text{ cm} \times 1.27\text{ cm} \times 0.089\text{ cm}$. The surfaces studied were EDM-cut, polished to mirror-finish, and oxidized (both polished and EDM-cut). Table 5.1 shows the experimental matrix for the oxidations in this study. Long-term oxidations were carried out using HZS 12/600 three-zone tube furnace (Carbolite Gero Ltd) as shown in Figure 3.2-a. The furnace has a uniformity of $\pm 5\text{ }^{\circ}\text{C}$ over a length of 500 mm to ensure a uniform temperature over the entire strip. Multiple strips were oxidized at the same time using a custom ground quartz boat. The heating rate of the furnace is $5\text{ }^{\circ}\text{C min}^{-1}$ to reduce breakage of process tube from thermal shock. To accelerate oxidation, samples already oxidized at 573 K for 1,000 h were oxidized further for additional 500 h at 773 K. This later oxidation resulted in spallation of the oxide layer and hence no emissivity data were obtained on such layers.

Once the oxidation was complete, five thermocouples were attached to the strips. The strips were washed to remove any contamination using a bath of acetone followed by a rinse of deionized water. The strip was secured within the sample holder, and each thermocouple is attached to its corresponding feed-through. Following ASTM C835-06, the chamber is first evacuated to an initial pressure of 5×10^{-2} torr and then heating is initiated using heating tape to help degassing of the system. Once the pressure reaches

1×10^{-5} torr, the degassing is complete. The test strip is resistively heated when the internal chamber temperature is about 295 K.

Surface roughness and oxide layers were analyzed after emissivity measurements. Roughness profiles for polished and EDM-cut strips were obtained by optical profilometry (Veeco Wyko NT-9100). Surface morphology and composition were determined with SEM equipped with EDS detector for X-Ray microanalysis. The strips were analyzed to observe the effect of oxide layer for various oxidation temperatures and times on the total hemispherical emissivity.

Table 5.1: *Experimental Matrix for A508/A533B Oxidation and Emissivity Measurements, and Results (we have also included results for polished and un-oxidized A508/A533B here).*

Condition	Oxidation Temperature (K)	Oxidation Time (h)	Temperature Range (K) for Emissivity Measurements	Measured Emissivity (approximate range)
Polished	None	None	552 – 1180	0.16 – 0.24
	573	10	545 – 815	0.18 – 0.28
		100	529 – 815	0.19 – 0.33
	773	10	429 – 739	0.72 – 0.81
EDM – Cut	None	None	464 – 845	0.51 – 0.54
	573	500	419 – 626	0.76 – 0.80
		1000	417 – 639	0.76 – 0.81
	773	500	429 – 657	0.71 – 0.81

5.3 Total Hemispherical Emissivity Measurements of A508/A533B:

Results & Discussion

The total hemispherical emissivity for EDM-cut and polished A508/A533B are given in Figure 5.1. The mirror-polished A508/A533B is considered to be the baseline case as the emissivity should mostly depend on its composition rather than topology. The total hemispherical emissivity of polished A508/A533B increased from 0.17 at 552 K to 0.24 at 1180 K. An inflection in the un-oxidized polished data occurs around 800 K where the emissivity is constant below and increases linearly at rate of $1.7 \times 10^{-4} \text{ K}^{-1}$ above 800 K, respectively. EDM-cut A508/A533B has a total hemispherical emissivity of 0.51 at 464 K to 0.54 at 845 K. The ASTM C835-06 requires the emissivity of a previously measured point not to exceed 2% of its original value. If so, the surface is considered to have changed during measurement. Both the polished and EDM-cut samples did not exceed this criterion and were with maximum electrical current allowed by the system (~ 100 Amps). No surface change occurred during measurements as warned by the testing standard. Two-dimensional maps of the roughness profiles of the EDM-cut and polished surfaces have been presented in Figures 5.2 and 5.3. The arithmetic average of the variation in height, R_a , and root mean square variation in height, R_q , are given in Table 5.2 measured at 5 different locations along the test section. The average values of R_a and R_q (root-mean-square) are also given in Table 5.2. The EDM-cut strip surface was ~ 171 times rougher than the polished surface with a fairly isotropic distribution of peaks and valleys as seen in Figure 5. 2- a. This surface texture will have an inherently higher emissivity through its increase absorptivity.

From Figure 5. 2- a and Table 5.2, the topology of the polished surface varies slowly as indicated by the large differences in R_a and R_q among the 5 different locations measured. The difference in roughness will affect the emissivity, but surface of the EDM-cut samples could be oxidized from the cutting process.

Figure 5. 1. Total hemispherical emissivity of A508/A533B steel after EDM cutting process and polished.

(b)

Figure 5.2. Optical profilometer scan of A508/A533B EDM cut :(a) 2D chromatic map of topography, and (b) extracted roughness profile along the x-direction as indicated in the two-dimensional map. Measurements were taken at the center of the test section (magnification:5.07 X, measurement mode: vertical scanning interferometer).

(a)

(b)

Figure 5.3. Optical profilometer scan of A508/A533B polished : (a) 2D chromatic map of topography, (b) extracted roughness profile along the x-direction as indicated in the two-dimensional map. Measurements were taken at the center of the test section (magnification: 5.07 X, measurement mode: vertical scanning interferometer).

Table 5.2 Surface roughness parameters at five different locations and their averages for EDM cut and polished A508/A533B.

Location	EDM cut		polished	
	Ra (μm)	Rq (μm)	Ra (μm)	Rq (μm)
1	3.95	4.88	28.57	34.15
2	4.02	4.91	25.14	30.89
3	3.53	4.32	17.26	21.56
4	4.12	5.06	19.72	24.63
5	3.99	4.85	24.54	30.24
Average	3.922	4.804	23.046	28.294

The effect of oxidation time and temperature on total hemispherical emissivity of polished strips are shown in Figures 5.4 and 5.5. Data for the polished and oxidized samples were reported up the temperature where the surface was stable under vacuum as per the ASTM C835-06. The emissivity of all the surfaces in Figure 5.4 approach the value of 0.16-0.17 at the lowest measured temperature. Oxidation of strips at 573 K has a pronounced effect on the rate of increase of emissivity with temperature. A straight line fit to the oxidized-polished strips emissivity values yields a slope of $3.2 \times 10^{-4} \text{ K}^{-1}$ and $7.1 \times 10^{-4} \text{ K}^{-1}$ for oxidation times of 10 h and 100 h, respectively. That is, the rate of increase in emissivity doubled with longer times. This implies that the difference in

emissivity may be greater at higher temperatures. There is a 21% difference in emissivity values at 815 K relating to oxidation times of 10 h and 100 h. Figures 5.6 and 5.7 show the electron micrographs and X-ray spectra of polished strips oxidized for 10 h and 100 h, respectively. The 10 h oxidized surface, Figure 5.6- a, appears relatively unchanged while the 100 h oxidized surface, Figure 5.7-a, appears roughened with the formation of nodules. Oxidation temperature at 10 h has a greater effect on the emissivity of polished A508/A533B.

Figure 5. 4. Total hemispherical emissivity of A508/A533B polished and oxidized strips in air at 573 K for 10 h and 100 h.

Figure 5. 5. Total hemispherical emissivity for oxidized A508/A533B for 10 h at different oxidation temperatures.

As seen in Figure 5.5, the total hemispherical emissivity increased from 0.74 to 0.81 in the temperature range of 462 K to 839 K for oxidized strips at 773 K for 10 h. For oxidation at 573 K for 10 h, the emissivity increased from 0.19 at 545 K to 0.28 at 820 K. SEM micrographs and EDS spectra for the 10 h oxidations at 573 K and 773 K are shown in Figures 5.6 and 5.8.

(a)

(b)

Figure 5. 6. SEM-EDS analysis of A508/A533B oxidized in air at 573 K for 10 h (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

(a)

Figure 5.7. SEM images and EDs for A508/A533B polished and oxidized in air at 573 K for 100 h (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

(a)

(b)

Figure 5. 8. SEM-EDS analysis of A508/A533B oxidized in air at 773 K for 10 h (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

The total hemispherical emissivity values for EDM cut strips oxidized in air at 573 K for 500 h and 1000 h are presented in Figure 5.9. Again, these samples reached a temperature where the surface changed. The total hemispherical emissivity for oxidized strips at 573 K for 500 h increased from 0.76 at 419 K to 0.80 at 625 K. Above 625 K the emissivity dropped but satisfied the retest criteria for the testing standard. No further significant increase in emissivity values for oxidized A508/A533B for 1000 h was observed. This slight change in emissivity values can be ascribed to low rate of oxide growth as oxidation time increased from 500 h to 1000 h at this low temperature. Figures 5.10, 5.11, and 5.12 show the electron micrographs and X-ray spectra of unoxidized EDM cut and oxidized EDM cut A508/A533B in air at 573 K for 500 and 1000 h respectively. The unoxidized EDM cut strip, Figure 5.10-a, appears roughened with the formation of nodules. Oxidation in air at 573 K for 500 h, Figure 5. 11-a shows numerous round nodules on the surface of oxidized EDM cut strips. The 1000 h oxidized surface, Figure 5.12-a, appears relatively unchanged from the 500 h oxidized surface.

Figure 5. 9. Total hemispherical emissivity of A508/A533B EDM oxidized and unoxidized strips.

(a)

(b)

Figure 5.10. SEM images and EDS for A508/A533B EDM cut (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

(a)

(b)

Figure 5.11. SEM images and EDS for A508/A533B oxidized in air at 573 K for 500 h (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

(a)

(b)

Figure 5.12. SEM images and EDS for A508/A533B oxidized in air at 573 K for 1000 h (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

The SEM-EDS results show an increase in oxygen content for polished and oxidized strips with increasing time, while the amount of oxygen for EDM cut oxidized strips at 500 h and 1000 h is approximately the same. This means that after 500 h, no more oxygen atoms can penetrate the A508/A533B substrate. In addition, copper appears in EDS analysis for unoxidized and oxidized EDM-cut strips. This can be ascribed to a recasting layer resulting from EDM cut (copper wires were used during the EDM cutting process). The semiquantitative results of the EDS analysis for selected elements are presented in Table 5.3. It can be clearly noticed from Table 5.3 that polished A508/A533B has no copper which gives an indication that recasting layer has been removed. The results show a high oxidation rate of A508/A533B at temperature 773 K. The spectral emissivity measurements of King et al.⁴⁶ showed emissivity values close to 0.9 at a wavelength range from 2 μm to 9 μm for oxidized A508/A533B at 873 K for 5 h. This is very close to the results of our present work for polished and oxidized A508/A533B at 773 K for 10 h.

Table 5.3 Semiquantitative Results of Oxygen and Selected Constituents of A508/A533B on Oxidized surface.

Oxidation		EDS Normalized Concentration (wt %)					
Temperature[K]	Time [h]	O	Fe	Mo	Mn	Cu	Zn
Polished							
573	10	4.39	91.25	0.32	0.77	--	--
573	100	9.25	85.38	0.31	1.48	--	--
773	10	25.82	64.57	0.15	1.31	--	--
EDM cut							
573	500	18.54	56.67	0.21	0.78	9.29	9.55
573	1000	19.98	53.72	0.16	0.61	7.38	9.39
EDM cut and Oxidized for 1000 h at 573 K							
773	500	16.67	22.65	1.69	0.97	51.07	0.55
EDM cut (unoxidized)							
--	--	5.9	68.95	0.21	2.46	10.62	4.23

To accelerate oxidation, oxidized samples for 1000 h at 573 K were oxidized again for 500 h at 773 K. After oxidation, the oxide layer detached from the substrate as can be seen in Figure 5.13. This can be ascribed to loss contact between the oxide layer and the metal substrate.⁵³ The total hemispherical emissivity measurements for oxidized EDM cut A508/A533B at 573 K and 773 K for 500 h are given in Figure 5.14. It can be noticed there is a slight change in emissivity values at temperature 600 K and up. SEM micrograph and EDS analysis were taken to analyze the detached oxide layer. The SEM micrograph and EDS results are given in Figure 5.15 showing two different phases of oxide layer.

Figure 5.13. A508/A533B strips after oxidation in air at 773 K for 500 h

Figure 5.14. Total hemispherical emissivity for A508/A533B EDM un-oxidized and EDM oxidized for 500 h at 573 K and 773 K.

(a)

(b)

(c)

Figure 5.15. SEM-EDS analysis of detached oxide layer for A508/A533B oxidized in air at 773 K for 10 h (a) backscattered electron micrograph (b) Secondary electron micrograph and (c) EDS X-ray spectrum.

5.4 Conclusion

Total hemispherical emissivity of A508/A533B has been measured for various surface conditions over the temperature ranges from 400 K to 1200 K. Polishing A508/A533B allowed the examination of oxidation duration and temperature without the effects of roughness and recasting layer from EDM cutting. Polished A508/A533B remained stable under vacuum up to 1200 K with an emissivity of 0.17 at 552 K to 0.24 at 1180 K. Low temperature oxidation of polished A508/A533B increased the rate of emissivity proportional to oxidation time. Oxidation temperature had significant impact on emissivity such that the values increased threefold when oxidation temperature increased from 573 K to 773 K for the same oxidation time (10 h). Cutting with wire EDM produced a rough surface with the presence of recast layer with an emissivity varying from 0.51 to 0.54 over the temperatures 464 K to 845 K. This surface could be close to actual surfaces of A508/A533B in nuclear plant applications where oxidation and contamination of the surface occur from manufacturing. Oxidation of EDM cut A508/A533B at 573 K for 500h, increased the emissivity from 0.75 at 418 K to 0.8 at 625 K with no further increase in emissivity for oxidation time of 1000 h. Thus, there could be a limiting value to the emissivity of A508/A533B for long-term exposure in low temperature environments like in LWRs. Attempts to accelerate the oxidation for further increase to the emissivity A508/A533B resulted in spallation of the oxide layer. Emissivity of the of already oxidized A508/A533B strips oxidized for an additional 500 h at 773 K had a similar emissivity for EDM cut A508/A533B oxidized at 573 K for 500 h. This could have implications in radiant

transfer of decay heat during an air ingress accident where the newly formed oxide layer fails limiting its emissivity to the condition it was prior to the accident. We have included a summary of the main results in Table 5.1.

CHAPTER 6 EMISSIVITY OF A387 GRADE 91 STEEL

6.1 Introduction

Ferritic steel alloys are an important part of materials, they have been developed for application as a structural material at elevated temperatures for power generation industries. Increasing new generation power plants life time operation has been led to modify 9% Cr steels with excellent combination of creep strength and ductility.^{54,55} A 387 Grade 91 is a candidate structural material for Very High Temperature Reactor (VHTR) pressure vessel due to a combination of high strength at high temperature and long thermal fatigue life.

For (VHTR) systems, it is important that the Reactor Pressure Vessel (RPV) has the ability to radiate heat during operations. Emissivity is a surface property that dictates the ability to radiate heat which is defined as the ratio of the radiance spectrum of material's surface to that of blackbody. A limited data exists on spectral and total hemispherical emissivity of A387 Grade 91. We reviewed these data in chapter 3. In this chapter, the total hemispherical emissivity values for A387 Grade 91 were measured in the temperature range of 400 K to 1048 K using ASTM standard C835-06. The effect of oxidation temperature and oxidation time were investigated.

6.2 Material and Sample Preparation

A387 Grade 91 steel was cut using wire electric discharge machining (EDM) from a 1.27 cm thick plate into test strips with size of 25.4 cm long, 1.25 cm width and 0.075 cm thick. Table 6.1 shows the composition of A387 Grade91.

Table 6.1 *Nominal composition of A387 Grade 91.*

Elements	Weight %
Fe	Balance
Cr	8.00 to 9.50
Mo	0.85 to 1.05
Ni (max)	0.4
Mn	0.30 to 0.60
Si	0.20 to 0.50
V	0.18 to 0.25
C	0.07 to 0.14
Nb	0.06 to 0.1
P (max)	0.02
Al (max)	0.02
S (max)	0.01
Ti (max)	0.01
Zr (max)	0.01

Prior to oxidation and emissivity measurements, the A387 Grade 91 strips were cleaned by a bath of acetone and rinse with deionized water and then they washed with ethanol and rinse with deionized water and left them dry in air. The studied surfaces were EDM cut, sandblasted with 320 grit size, and oxidized (both EDM cut and sandblasted samples). Surface roughness and oxide layers were analyzed after emissivity measurements. Roughness profiles for polished and EDM cut A387 Grade 91 were obtained by optical profilometry (Veeco Wyko NT-9100). Surface morphology and composition were done with SEM equipped with EDS detector for X-Ray microanalysis. The A387 Grade 91 samples were analyzed to observe the effect of oxide layer for various oxidation temperatures and times on the total hemispherical emissivity. The experimental matrix is illustrated in Table 6.2.

The short - term oxidation were obtained using HZS 12/600 three zone tube furnace (Carbolite Gero Ltd) as shown in Figure 3.3-b. To ensure the oxidation has not occurred during furnace heating and cooling, we used vacuum pump to evacuate the furnace under vacuum pressure between 100- 300 mtorr (see section 3.3.2 for more details). The air flow rate used during oxidation was 150 mL min^{-1} . Five type K thermocouples were attached to the strip after oxidation to measure the temperature along the sample strip.

Table 6.2: *Experimental Matrix for A387 Grade 91 Oxidation and Emissivity Measurements, and Results (we have also included results for polished and un-oxidized A387 Grade 91 here).*

Condition	Oxidation Temperature (K)	Oxidation Time (h)	Temperature Range (K) for Emissivity Measurements	Measured Emissivity (approximate range)
EDM cut	None	None	444-1026	0.57-0.65
	873	1	419-831	0.75-0.8
		3	420-898	0.74-0.81
		5	421-885	0.73-0.82
	1023	1	430-942	0.69-0.81
		3	429-920	0.68-0.8
		5	427-941	0.71-0.81
Sandblasted 320 grit size	None	None	464-1048	0.49-0.57
	1023	5	440-945	0.63-0.74

In this chapter, the effect of oxidation temperature and oxidation time on total hemispherical emissivity of A387 Grade 91 was investigated. The surface roughness was measured using optical profilometry and oxide layer was analyzed using scanning electron microscope (SEM). Standard ASTM C835-06²² calorimetric was used to measure total hemispherical emissivity of A387 Grade 91 ferritic steel.

6.3 Total Hemispherical Emissivity Measurements of A387 Grade 91:

Results and Discussion

The total hemispherical emissivity of A387 Grade 91 EDM cut as a function of temperature is shown in Figure 6.1. The total hemispherical emissivity data from Azmeh et al.³⁵ for mild-abrasive A387 Grade 91 are also included. The emissivity of A387 Grade 91 EDM cut has increased from 0.57 to 0.65 in range of temperatures between 444 K and 1026 K.

Figure 6.1. Total hemispherical emissivity of A387 Grade 91 steel after EDM cut compared to Azmeh et al. (2016) data for A387 Grade 91 mild-abrasive.

The total hemispherical emissivity values for A387 Grade91 EDM cut were higher than the emissivity values of mild-abrasive A387 Grade 91 in Azmeh et al.³⁵ work. This can be ascribed to oxidation occurring during EDM cutting method from the heat generated. Figure 6.2 shows the surface morphology and EDS spectra to illustrate the presence of oxygen after EDM cut process. The topography of roughened surfaces and roughness profiles of the EDM cut is presented in Figure 6. 3. The arithmetic average of the variation in height, R_a , and root mean square variation in height, R_q , are given in Table 6.3 measured at 5 different locations along the test section. The average values of R_a and R_q (root-mean-square) are also given in Table 6.3.

Table 6.3 Surface roughness parameters at five different locations and their averages for EDM cut and sandblasted A387 Grade 91.

Location	EDM cut (un-oxidized)		Sandblasted with 320 grit size (un-oxidized)	
	R_a (μm)	R_q (μm)	R_a (μm)	R_q (μm)
1	3.93	3.66	2.10	2.65
2	3.33	4.16	2.03	2.55
3	3.68	4.55	2.04	2.55
4	3.44	4.27	1.94	2.46
5	3.08	3.83	2.32	2.94
Average	3.64	4.10	2.09	2.63

(a)

(b)

Figure 6.2 SEM-EDS analysis of EDM cut A387 Grade 91; (a) Secondary electron micrograph and (b) EDS X-ray spectrum

(a)

(b)

Figure 6.3. Optical profilometer scan of EDM cut A 387 Grade 91: (a) 2D chromatic map of topography, (b) extracted roughness profile along the x-direction as indicated in the two-dimensional map. Measurements were taken at the center of the test section (magnification:5.07 X, measurement mode: vertical scanning interferometer).

To study the effect of surface roughness on total hemispherical emissivity of A387 Grade 91, strips were sandblasted using aluminum oxide beads with 320 grit size. The total hemispherical emissivity values are given in Figure 6.4. The emissivity data for Azmeh et al.³⁵ for sandblasted surfaces with 60 and 120 grit size are included. The emissivity values of sandblasted surface with 320 grit size increased from 0.49 at 464 K to 0.57 at 1048 K. From Figure 6.4, above 800 K, a straight line fit to the sandblasted A387 Grade 91 with 320 grit size yields a slope of $3.47 \times 10^{-4} \text{ K}^{-1}$.

Figure 6.4 Total hemispherical emissivity of A387 Grade 91 sandblasted with 320 grit size and compared to Azmeh et al. (2016) data for sandblasted A387 Grade 91 with 60 and 120 grit size.

Figure 6.5. Optical profilometer scan of A 387 Grade 91 sandblasted with 320 grit size (a) 2D chromatic map of topography, (b) extracted roughness profile along the x-direction as indicated in the two-dimensional map. Measurements were taken at the center of the test section (magnification: 5.07 X, measurement mode: vertical scanning interferometer).

As illustrated in Figure 6.4, the total hemispherical emissivity of sandblasted surfaces increased with finer grit size used. The topography of roughened surface and roughness profile of the sandblasted A387 Grade 91 with 320 grit size is presented in Figure 6.5. The arithmetic average of the variation in height, R_a , and root mean square variation in height, R_q , are given in Table 6.3. Data for EDM cut and sandblasted test strips were reported up the temperature where the surface was stable under vacuum as per ASTM standard C835-06.

The effect of oxidation time and oxidation temperature on total hemispherical emissivity of A387 Grade 91 is given in figures 6.6 through 6.10. Figures 6.6 and 6.7 represent the total hemispherical emissivity data for EDM cut oxidized in air at temperatures 873 K and 1023 K for 1 h, 3 h, and 5 h respectively. In general, the total hemispherical emissivity strongly increased with oxidation at two different temperatures 873 K and 1023 K compared to those un-oxidized. The total hemispherical emissivity of A387 Grade 91 EDM cut and oxidized for 1 h at temperature 873 K varied from 0.75 at 420 K to 0.8 at 831 K, while the emissivity of A387 Grade 91 EDM cut and oxidized strips at 1023 K for 1 h increased from 0.69 to 0.81 in temperature range between 430 K to 942 K. Oxidation of A387 Grade 91 in air at 873 K and 1023 K did not result in significant difference in emissivity among the various sample strips for oxidation durations of 1 h, 3 h, and 5 h.

Figure 6.6 Total hemispherical emissivity of oxidized A387 Grade 91 in air at 873 K for 1 h, 3 h, and 5 h.

Figure 6.7. Total hemispherical emissivity of oxidized A387 Grade 91 in air at 1023 K for 1 h, 3 h, and 5 h.

The effect of oxidation temperature on total hemispherical emissivity of A387 Grade 91 is shown in Figures 6.8, 6.9, and 6.10. The expectation is as the temperature increases, the oxidation levels increase, resulting in increased emissivity values. The oxide layer characteristic of A 387 Grade 91 test strips processed at different oxidation temperatures (873 K and 1023 K) were quite different. For the oxidation temperature 873 K, the oxide layer formed on the A387 Grade 91 surface is most likely copper oxide due to high amount of copper appears in EDS analysis results as shown in Table 6.4.

Figure 6.8. Total hemispherical emissivity of oxidized A387 Grade 91 in air for 1h at 873 K and 1023 K.

Figure 6.9. Total hemispherical emissivity of oxidized A387 Grade 91 in air for 3 h at 873 K and 1023 K.

Figure 6.10. Total hemispherical emissivity of oxidized A387 Grade 91 in air for 5h at 873 K and 1023 K.

The semiquantitative results of the X-ray for selected elements are presented in Table 6.4. The data appear to show that the oxygen concentration is inconsistent with oxidation time increase at 873 K. However, the oxygen concentration increased with oxidation time at 1023 K. This is because each test strip is different, and the sampling points are different. The semiquantitative results also showed the presence of high copper amounts, that were a result of recasting layer formed during EDM cutting process.

Table 6.4 Semiquantitative Results of Oxygen and Selected Constituents of A387 Grade 91 on oxidized surface and un-oxidized surface.

Oxidation Time	EDS Normalized Concentration (wt %)					
	O	Cr	Mo	Fe	Cu	Zn
	After EDM cut					
	8.09	4.34	0.35	52.24	14.83	0.35
	EDM cut oxidized at 873 K					
1 h	20.37	0.78	0.02	43.11	30.13	2.61
3 h	17.5	1.35	0.04	45.72	30.95	2.29
5 h	17.8	1.29	0.05	39.73	36.26	2.44
	EDM cut oxidized at 1023 K					
1 h	19.15	1.06	0.105	46.75	30.75	--
3 h	22.42	10.14	0.23	54.65	10.74	2.11
5 h	25.59	5.89	0.11	52.76	12.35	0.63
	SB 320 grit size(un-oxidized)					
	3.92	8.4	0.48	78.73	3.09	--
	SB 320 grit size(oxidized)					
5 h	31.62	9.2	0.36	35.89	0.84	--

Therefore, the oxide layer at 873 K is less condense and it has some pores allowing for more oxidation as shown in Figures 6.11,6.12, and 6.13. For oxidation temperature 1023 K, the total hemispherical emissivity values are lower. The reason may be explained that at high temperature, the oxide layer formed on A387 Grade 91 surface is chromium oxide, which is considered as a protective layer inhibiting further oxidation.

In addition, at high temperature 1023 K, the oxide layer was compact, and its density was correspondingly large. Therefore, the microstructure was homogeneous, and the crystals gradually grow with oxidation time as can be seen in figures 6.14, 6.15, and 6.16.

(a)

(b)

Figure 6.11 SEM–EDS analysis of A387 Grade 91 oxidized at 873 K for 1 h: (a) Secondary electron micrograph of two different magnifications, and (b) EDS X-ray spectrum.

(a)

(b)

Figure 6.12 SEM–EDS analysis of A387 Grade 91 oxidized at 873 K for 3 h: (a) Secondary electron micrograph of two different magnifications, and (b) EDS X-ray spectrum.

(a)

(b)

Figure 6.13 SEM–EDS analysis of A387 Grade 91 oxidized at 873 K for 5 h: (a) Secondary electron micrograph of two different magnifications, and (b) EDS X-ray spectrum.

(a)

(b)

Figure 6.14 SEM–EDS analysis of A387 Grade 91 oxidized at 1023 K for 1 h: (a) Secondary electron micrograph of two different magnifications, and (b) EDS X-ray spectrum.

(a)

(b)

Figure 6.15 SEM–EDS analysis of A387 Grade 91 oxidized at 1023 K for 3 h: (a) Secondary electron micrograph of two different magnifications, and (b) EDS X-ray spectrum.

(a)

(b)

Figure 6.16 SEM–EDS analysis of A387 Grade 91 oxidized at 1023 K for 5 h: (a) Secondary electron micrograph of two different magnifications, and (b) EDS X-ray spectrum.

The total hemispherical emissivity data of A387 Grade 91 sandblasted with 320 grit, un-oxidized and oxidized in air at 1023 K for 5 h are shown in Figure 6.17. The total hemispherical emissivity of sandblasted A387 Grade 91 oxidized in air for 5 h increased from 0.63 at 440 K to 0.74 at 945 K, while the emissivity of un-oxidized strips increased from 0.49 to 0.57 in range of temperature between 464 K and 1048 K. The emissivity values increased with oxidation. A fit straight line sandblasted A387 Grade 91 above 800 K yields a slope of $1.9 \times 10^{-4} \text{ K}^{-1}$ for un-oxidizes data and $1.49 \times 10^{-4} \text{ K}^{-1}$. There is a 29.8% difference in emissivity from oxidized and un-oxidized sandblasted A387 Grad 91 at high temperature.

Figures 6.18 and 6.19 show the electron micrographs and X-ray spectra for A 387 Grade 91 sandblasted strips with 320 grit size unoxidized and sandblasted with 320 grit size and oxidized respectively. Figure 6.19-a shows numerous round nodules on the surface of sandblasted and oxidized A387 Grade 91.

Figure 6.17. Total hemispherical of A387 Grade 91 sandblasted with 320 grit size and oxidized at 1023 K for 5 h and unoxidized.

(a)

(b)

Figure 6.18 SEM and EDS analysis for A387 Grade 91 sandblasted with 320 grit unoxidized (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

(a)

(b)

Figure 6.19 SEM and EDS analysis for A387 Grade 91 sandblasted with 320 grit oxidized at 1023 K for 5 h (a) Secondary electron micrograph and (b) EDS X-ray spectrum.

6.4 Conclusion

The total hemispherical emissivity data for A387 Grade 91 were reported. The emissivity measurements were taken in the range of temperatures between 440 K to 1050 K. The total hemispherical emissivity for oxidized test strips has significantly increased. The emissivity of A387 Grade 91 oxidized at 873 K increased from 0.75 at 420 K to 0.8 at 831 K, while the emissivity of oxidized A387 Grade 91 at 1023 K increased from 0.69 at 429 K to 0.8 at 889 K. No further significant difference in emissivity values was noticed with increasing oxidation time. Increasing oxidation temperature from 873 K to 1023 K resulted in decreasing emissivity particularly at lower temperatures. The reason may be explained to two different oxide layers forming on A387 Grade 91 surface at two different temperatures. The total hemispherical emissivity of sandblasted and oxidized A387 Grade 91 in air at 1023 K for 5 h increased from 0.63 at 440 K to 0.74 at 945 K whereas the emissivity of unoxidized sandblasted A387 Grade 91 has increased from 0.49 to 0.57 in the range of temperatures from 464 K to 1048 K.

CHAPTER 7 CONCLUSION AND FUTUER WORK

7.1 Conclusion

In this work, total hemispherical emissivity of three candidate material: SS316L, A508/A533B, and A387 Grade 91 has been measured for various surface oxidations. The total hemispherical emissivity for SS316L measurements were taken in the range of temperature between 430 K to 1100 K. SS316L test strips were oxidized in air at constant low temperatures 573 K up to 3000 h. The emissivity significantly increased for oxidized samples at 573 K for 500 h compared to as-received strips (unoxidized). No further increase in emissivity values was observed with increasing oxidation time. This can be attributed to protective chromium oxide layer formed on SS316L surface.

A508/A533B samples were exposed to either polishing or oxidation or a combination of polishing and oxidation. Polished strips have low emissivity values while the emissivity of polished and oxidized samples was significantly high, particularly with increasing oxidation temperature. The emissivity of EDM cut and oxidized EDM cut strips increased with temperature increase. The results showed that oxidation temperature has a stronger effect than the oxidation time.

The total hemispherical emissivity for A387 Grade 91 a candidate material for RPV was measured in temperature range from 440 k to 1050 K. The A387 Grade 91 surfaces were exposed to air at temperatures 873 K and 1023 K. The emissivity behavior was the same for the two oxidation temperatures, increasing emissivity with increasing

temperature. At high temperature 1023 K, the total hemispherical emissivity values were lower than those at 573 K for the same oxidation time. The strips were not oxidized, sandblasted with 320 grit size. The emissivity of unoxidized sandblasted strips increased from 0.49 at 444 K to 0.65 at 941 K. The emissivity of oxidized sandblasted surfaces from 0.63 at 440 K to 0.74 at 945 K.

In conclusion, both surface oxidation and surface roughness have affected the total hemispherical emissivity of the three candidate materials. Oxidation has the utmost significant impact on total hemispherical emissivity, particularly oxidation temperature.

7.2 Future work

- Oxidized the three candidate materials for close intervals and measure the oxide layer thickness to find the optimum thickness that contributes to produce high emissivity.
- Simulate the parameters that affect the total hemispherical emissivity and optimize them using COMSOL software. That will help reducing time and number of experiments to reach the desire emissivity in nuclear reactors.

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VITA

Faten Al Zubaidi

Faten Al-Zubaidi was born on November 4, 1972, in Iraq. She grew in Baghdad and graduated from the University of Technology-Baghdad. In 1994, she earned her bachelor's degree in mechanical engineering from the University of Technology. In 2002, she earned her master's degree at University of Technology-Baghdad. She applied for a doctoral scholarship in 2011, and finally got accepted in 2013. She came with her family and has studied and prepared to present her dissertation defense. During the past five years, she has experienced many hardships that she overcame with the help of her family and friends. She earned a second master's degree at the University of Missouri in May 2017.