2005

Study of hideout and hideout return in a simulated steam generator

Nondha Sittiratanarangsee
San Jose State University

Follow this and additional works at: https://scholarworks.sjsu.edu/etd_theses

Recommended Citation

Sittiratanarangsee, Nondha, "Study of hideout and hideout return in a simulated steam generator" (2005). *Master's Theses*. 2727.

DOI: <https://doi.org/10.31979/etd.mef9-vfzy>
https://scholarworks.sjsu.edu/etd_theses/2727

This Thesis is brought to you for free and open access by the Master's Theses and Graduate Research at SJSU ScholarWorks. It has been accepted for inclusion in Master's Theses by an authorized administrator of SJSU ScholarWorks. For more information, please contact scholarworks@sjsu.edu.

**STUDY OF HIDEOUT AND HIDEOUT RETURN IN
A SIMULATED STEAM GENERATOR**

A Thesis

Presented to

The Faculty of the Department of Chemical Engineering

San Jose State University

In Partial Fulfillment

of the Requirements for the Degree

Master of Science

by

Nondha Sittiratanarangsee

May 2005

UMI Number: 1427182

INFORMATION TO USERS

The quality of this reproduction is dependent upon the quality of the copy submitted. Broken or indistinct print, colored or poor quality illustrations and photographs, print bleed-through, substandard margins, and improper alignment can adversely affect reproduction.

In the unlikely event that the author did not send a complete manuscript and there are missing pages, these will be noted. Also, if unauthorized copyright material had to be removed, a note will indicate the deletion.

UMI Microform 1427182

Copyright 2005 by ProQuest Information and Learning Company.

All rights reserved. This microform edition is protected against unauthorized copying under Title 17, United States Code.

ProQuest Information and Learning Company
300 North Zeeb Road
P.O. Box 1346
Ann Arbor, MI 48106-1346

© 2005

Nondha Sittiratanarangsee

ALL RIGHTS RESERVED

APPROVED FOR THE DEPARTMENT OF CHEMICAL ENGINEERING

3-16-2005

Dr. Melanie A. McNeil

Dr. Richard W. Chung

Dr. Peter J. Millett, iSagacity

APPROVED FOR THE UNIVERSITY

ABSTRACT

STUDY OF HIDEOUT AND HIDEOUT RETURN IN A SIMULATED STEAM GENERATOR

by Nondha Sittiratanarangsee

Corrosion problems in steam generators have forced the early shutdown of nuclear power plants in the United States. In this study, a carbon-packed crevice situated in a high-temperature, high-pressure autoclave was used to simulate a partially fouled crevice of the type that causes corrosion in steam generators. By using a 60 ppm NaCl solution to represent typical power plant feedwater impurities, a hideout and hideout return corrosion process was used to study the autoclave performance. The autoclave was operated below the water saturation point of 510 °F and 871 psig. When heat was added to the carbon fiber-packed crevice, the solution temperature was elevated above the saturation point in the crevice creating accumulation (hideout). Once the crevice was saturated with NaCl, heat was removed causing the accumulated impurity to leave (hideout return). The results of the experiments performed were analyzed to verify performance of the autoclave.

ACKNOWLEDGEMENTS

I would like to thank my parents who always support me, both financially and mentally. I would like to thank Dr. Melanie McNeil for accepting me to study at San Jose State University. Dr. McNeil has always been a good counselor in academics and for other university issues. Dr. McNeil is also my thesis advisor who always gave good advice and helped me overcome all the obstacles.

TABLE OF CONTENTS

	page
CHAPTER ONE INTRODUCTION.....	1
1.1 BASIC OPERATION OF PRESSURIZED WATER REACTOR.....	4
1.2 CORROSION PROCESS.....	5
1.3 PREVENTING STEAM GENERATOR TUBE FAILURES.....	7
CHAPTER TWO LITERATURE REVIEW.....	9
2.1 CORROSION MITIGATION HISTORY.....	9
2.2 AUTOCLAVES AS SG SIMULATORS.....	10
2.3 AUTOCLAVE CONCEPT.....	11
2.4 DRY OUT DETECTION.....	18
2.5 MEASUREMENT TECHNIQUES.....	21
CHAPTER THREE EXPERIMENTAL DESIGN AND PROCEDURES.....	22
3.1 OBJECTIVES.....	22
3.2 EXPERIMENTAL METHODS.....	22
3.2.1 Feedwater System.....	22
3.2.2 Crevice.....	26
3.2.3 Autoclave.....	31
3.3 MEASUREMENT AND CONTROL OF PARAMETERS.....	33
3.3.1 Temperature.....	33
3.3.2 Pressure and Flow.....	33
3.3.3 pH.....	34
3.3.4 Conductivity.....	34
3.3.5 Data Collecting System.....	34
3.4 DESIGN OF EXPERIMENT.....	35

	page
3.5 THE EXPERIMENTAL SETUP.....	36
3.6 ANALYSIS OF DATA.....	36
CHAPTER FOUR RESULTS AND DISCUSSION.....	38
4.1 RECOMMISSION AND VERIFICATION.....	38
4.1.1 Diaphragm Pump.....	38
4.1.2 Preheater.....	38
4.1.3 Measurement Equipment Exit Line Modification.....	39
4.1.4 pH Meter.....	40
4.1.5 Precision Balance.....	40
4.1.6 Conductivity Meter.....	41
4.1.7 Thermocouples.....	43
4.2 EXPERIMENT RESULTS.....	44
4.2.1 Three Experiments with 30 Percent Porosity.....	44
4.2.2 Baseline Experiment.....	47
4.3 INVESTIGATING UNDESIRE HIDEOUT EXPERIMENTS.....	50
4.3.1 Old Surface-Corroded Clamshell as the Source of Hideout.....	50
4.3.2 Autoclave Lid as the Source of Hideout.....	51
4.3.3 Precipitation as a Cause of NaCl Disappearance and Reappearance.....	53
4.3.4 Low Flow as a Cause of NaCl Disappearance and Reappearance.....	56
CHAPTER FIVE FUTURE WORK.....	57
CHAPTER SIX CONCLUSIONS.....	58
REFERENCES.....	59
APPENDIX A STANDARD OPERATING PROCEDURES.....	61
APPENDIX B START UP AND SHUTDOWN PROCEDURE FOR THE SIMULATED STEAM GENERATOR.....	91

	page
APPENDIX C MISCELLANEOUS PROCEDURES.....	98
APPENDIX D ELECTRICAL DIAGRAM OF HEATERS.....	105
APPENDIX E TROUBLESHOOTING.....	108
APPENDIX F DATA ACQUISITION DIAGRAMS.....	110
APPENDIX G CHEMICALS AND EQUIPMENT.....	122
APPENDIX H ADDITIONAL EXPERIMENTAL RESULTS.....	123

LIST OF FIGURES

	page
Figure 1. Types of corrosion in nuclear power plant steam generators.....	3
Figure 2. A pressurized water reactor power generation cycle.....	5
Figure 3. Inside a steam generator.....	6
Figure 4. Tube support plate setup by Campan.....	12
Figure 5. Experimental result by Rogers shows hideout and hideout return.....	13
Figure 6. Accumulation of sodium in the crevice during hideout phase, compared between 5 and 50 ppm of sodium in the bulk water	14
Figure 7. PWR support crevice experimental setup by Millett.....	16
Figure 8. Steam blanket temperature distribution in a carbon fiber-packed crevice.....	20
Figure 9. Flow diagram of the experimental system.....	25
Figure 10. Cross-section of the clamshell.....	27
Figure 11. Top view and side view of half of a clamshell.....	28
Figure 12. Radial positions of each thermocouple.....	30
Figure 13. Steam blanket temperature distribution in a carbon fiber-packed crevice.....	31
Figure 14. Experimental result by Rogers shows hideout and hideout return.....	37
Figure 15. Old measurement equipment exit line.....	39
Figure 16. Modified measurement equipment exit line.....	40
Figure 17. Conductivity calibration curve.....	42
Figure 18. Hideout and hideout return result.....	45
Figure 19. Hideout and hideout return confirmation experiment.....	46
Figure 20. Hideout and hideout return from the other confirmation experiment.....	47

	page
Figure 21. Baseline experiment. No packing, 60 ppm NaCl and 150 watts.....	48
Figure 22. Baseline experiment, 0% packed crevice and 150 watts by Rogers	49
Figure 23. Baseline experiment. No clamshell, 60 ppm NaCl and 150 watts.....	51
Figure 24. Baseline experiment result: no-clamshell, 60 ppm NaCl and 150 watts when the eutectic salt level is above the crevice tube heater cartridge....	52
Figure 25. Pressure and temperature correlation.....	54

LIST OF TABLES

	page
Table 1. Experimental Matrix.....	35
Table 2. pH Meter Verification.....	40
Table 3. Precision Balance Verification.....	41
Table 4. Conductivity Meters Reading and Standard Solutions Correlation.....	41
Table 5. Crevice Tube Heater #1 Temperature Reading.....	43
Table 6. Crevice Tube Heater #2 Temperature Reading.....	43
Table 7. Eutectic Salt Temperature Reading.....	43
Table 8. Experimental Runs.....	45
Table 9. Summary of Total Accumulation for NaCl+DI Solution with 30% Packing Porosity and 150 Watts at the Superheat Phase.....	47
Table 10. Baseline Total Accumulation NaCl 60 ppm and 0% Packed Crevice...	48
Table 11. Baseline Total Accumulation NaCl 60 ppm. Decrease Eutectic Salt Level.....	52
Table 12. Summary of Experimental Results.....	53
Table 13. Pressure Test Experiment.....	55
Table 14. Future Maintenance.....	57

CHAPTER ONE

INTRODUCTION

Nuclear power has become a major power source in the United States over the past few decades. Nuclear power plants have expanded rapidly since 1973 and currently generate 20 percent of the electricity in the United States [3]. Generally, a steam generator (SG) in a nuclear power plant is designed to endure power cycle operation for 40 years. However, the SGs in many nuclear power plants have experienced corrosion, which leads to failure of the SGs, and requires replacement of the whole SG system earlier than its life expectancy, sometimes only 8-10 years after commissioning [2]. Corrosion in the heat exchanger tube of the steam generator in a nuclear power plant is found to be the leading cause of failure. As a result, several research studies on corrosion have been conducted to prolong the life of nuclear power plants.

In 1983, 47 nuclear power plants were shut down due to corroded SG tubes. Also in 1984, 63 nuclear power plants were shut down due to many types of SG corruptions. Intergranular stress corrosion cracking* (IGSCC) and intergranular attack** (IGA) were found to be the leading causes of the failures [6]. Power plant shutdown requires a considerable amount of time to find the causes of corrosion and maintain or replace power available to both industry and consumers. In short, unplanned shutdown of power plants has greatly impacted the economics for both power plants companies and consumers. In some cases, the tube failure has required replacement of the whole SG,

* Cracking in metal tube due to the stress from the manufacturing.

** Cracking in metal tube's grain boundary.

which costs around \$100 million to \$200 million, and in the worst case, the plant was permanently shut down [2].

A summary of the various corrosion types, which occurs in SGs, is provided in Figure 1.

Figure 1. Types of corrosion in nuclear power plant steam generators [2].

In an effort to lower the number of these unplanned shutdowns, many researchers have intensively studied the causes of corrosion in the crevices of power plant SGs. Steam generator simulators (autoclaves) have been developed over the past decades and have been used as a tool for understanding the corrosion process and how to mitigate corrosion problems. Current corrosion problems have been found to occur in the low flow and high heat flux regions of the steam generator such as gaps between tubes and tube support plates (TSP). These gaps are also called “crevices.”

1.1 Basic Operation of Pressurized Water Reactor

Most of the nuclear power plants in the United States use pressurized water reactors (PWR). Figure 2 illustrates the normal operation cycle of PWRs. PWR nuclear power plants use the heat from nuclear reaction to transfer energy to the fluid under pressure so that the fluid remains liquid. This heated liquid inside the SG tube bundle (primary loop) transfers heat to bulk water in the SG (secondary loop). The SG consists of bundles of metal tubes that are used to transfer the water. Tube support plates hold the bundles together. The heated water evaporates into powerful steam, which will be used to drive the turbine and generate electricity.

Figure 2. A pressurized water reactor power generation cycle [7].

1.2 Corrosion Process

In the steam generation process for PWRs, feedwater receives a heat flux gradient across the boiler tubes and then evaporates; the steam has little solubility for trace amounts of impurities. Although impurities in the feedwater is on the scale of parts per million (ppm) or parts per billion (ppb), impurities can accumulate through precipitation after many cycles of steam generation at high temperature and pressure. The phenomena of impurity accumulation can be detected at the SG outlet. When the outlet concentration of impurity is lower than the inlet, it indicates that the impurities have accumulated in the steam generator. The disappearance of impurity is called the “hideout” phenomenon. The impurities in the SG react with the tube and the tube support plate material and form the corrosion products. When the power plant shuts down, generally for maintenance,

noticeable amounts of impurities are detected. The phenomenon of impurities being released after heat flux is removed or reduced from the SG is called “hideout return.”

Inside small ceramic pellets encased in long metal rods

Figure 3. Inside a steam generator [7].

The corrosion is likely to take place in the low flow or stagnant region of the SG. The SG consists of many tubes, which are held together by support plates. The annular crevices between the tubes and the support plates are stagnant regions. The cross section of the SG, in Figure 3, shows the position of bundles of SG tubes and the tube support plates that hold these tubes together. When the bulk water receives the heat from the SG tubes, at the SG tubes outer surface, water evaporates rapidly. At the beginning phase of SG operation, nucleate boiling* occurs at the SG tubes’ heat transfer interface. After the nucleate boiling phase, evaporation near the low-flow, high-heat flux region creates a thin layer of steam, called a “steam blanket” at the surface of the crevices. This phenomenon is called “dry out.” The steam blanket accelerates the accumulation process of impurities

* Boiling occurring from superheating crevice.

and thus accelerates the SG corrosion process. Many types of corrosion in Figure 1 can occur at the crevices and once corrosion starts, it will propagate and cause plugging or leakage, which will eventually lead to tube failure in the SG [2].

1.3 Preventing Steam Generator Tube Failures

Some methods have been proposed to reduce power plants' downtime. For example, some SGs were designed with 10-20 percent more heat transfer area than usual to increase the life of the SG before maintenance. However, when 10-20 percent of these excess tubes are plugged or corroded, the SG still requires overhauling [2].

Another way to extend the SG's operational life in a nuclear power plant is to change the tube material. Most of the PWRs in the United States use mill-annealed Inconel alloy 600; many plants have replaced the whole SG with Inconel alloy 690 or Inconel alloy 800, which is more resistant to corrosion. According to the hot-acid-test by Berry [21], Inconel alloy 800 has 5 times more resistance than Inconel alloy 600. However, the replacement cost of SGs is approximately 100-200 million US dollars per unit. Due to the cost, many times, even when it is found that corrosion is occurring in the SG, or the heat exchange rate of the SG drops significantly, nuclear power plant managers face a tough decision whether or not to continue operation or replace the corroded SG immediately. Realistically, replacing the SG is usually done when the power plant has enough financial resources.

In some cases, corroded SGs are often required to operate until the company has enough money to replace the new SG unit, even if it means that the nuclear power plant is

taking the risk of SG tube failure problems. The best way to mitigate the SGs' corrosion problem is to understand the causes of corrosion processes [2].

Many researchers use autoclaves, which imitate the phenomena in the SG in a PWR nuclear power plant but on a much faster time scale. The autoclaves have been used as a tool to find the way to mitigate the corrosion problem in the real SGs.

CHAPTER TWO

LITERATURE REVIEW

2.1 Corrosion Mitigation History

Researchers have conducted several research studies finding ways to minimize tube failure problems. Since most nuclear power plants in the United States use PWRs, researchers have focused their study on corrosion in PWRs [15]. PWRs typically operate at a temperature range of 500 °F (260 °C) to 608 °F (320 °C) while the pressurized water remains liquid below the saturation point^{*} in the secondary loop [17].

Early nuclear power plants' SGs use mill-annealed alloy 600 as their material of construction. The early SGs experienced severe wastage corrosion problems^{**}. In order to solve this problem, researchers introduced all-volatile treatment (AVT), which uses oxygen scavengers and ammonia in demineralized water^{***} (DI water) to control the pH combined with alloy 690 or 800 material to replace alloy 600 in the SG at the appropriate time (depending on financial demands and engineering practice). These changes significantly lessened the corrosion problems [2, 17].

Although researchers successfully solved the original corrosion problem using the AVT program, other types of corrosion problems still exist. In the early 1990s, many nuclear power plants' SG experienced secondary-side stress corrosion cracking. The new types of corrosion that power plants are currently facing are intergranular attack (IGA)

^{*} Boiling point of water at specific temperature and pressure.

^{**} Tube thinning.

^{***} Very pure water with conductivity less than 10 µs/cm.

and intergranular stress corrosion cracking (IGSCC). In IGA, the grain boundary of the tube materials is chemically attacked. In IGSCC, the tube, which has received relatively high stress from the tube fabrication process, can have cracking along the grain boundary. Researchers are currently studying ways to reduce IGA and IGSCC problems; for example, controlling the ion concentration, which directly affects the pH in the crevices that could lessen IGA and IGSCC.

2.2 Autoclaves as SG Simulators

In nuclear power plants' SGs, when the heat loads decrease, or the plants shut down, some of the corrosion products are released from the crevices (hideout return). Analysis of these corrosion products allows researchers to understand the corrosion mechanism; however, nuclear power plants rarely shut down, sometimes only once or twice a year [13]. Measuring the *in situ* condition in real SGs is not practical. Without frequent analysis, it is difficult to study corrosion in the SG. Researchers cannot wait up to 8 to 10 years just for the corrosion to occur. As a result, numerous autoclaves have been constructed to simulate corrosion on a much faster time scale. In addition, mathematical models have been developed in the past couple of decades to describe transport processes in the SGs. These simulators and models allow engineers to understand the corrosion mechanisms and important parameters in order to find the appropriate way to mitigate the corrosion problem in the real nuclear power plants' SGs.

2.3 Autoclave Concept

Due to the complexity of the corrosion processes and water chemistry in the bulk water, the process needs to be simplified for the simulation study on a laboratory scale and in such a short time frame.

Sodium chloride (NaCl) solution is often selected to represent impurities, as it has the largest proportion of contamination in the feedwater.

In the study by Campan et al., two loops of water were set up in a column, the primary loop and the secondary loop as shown in Figure 4. The primary circuit represented the tube-side pressurized liquid while the secondary loop water represented the water-steam generation side. A tube support plate was attached to simulate the tube sheet crevices. The tube sheet crevices were pre-packed with magnetite [8].

The tube support plate's crevice fouling process, which can lead to corrosion, normally takes many years to occur. In order to simulate the fouling process in the practical experiment time frame of a few days, packing material was put in the crevice to accelerate the fouling process. In one of the research studies, Mann and Castle put carbon fiber as their packing material in their crevice [19].

Figure 4. Tube support plate setup by Campan [8].

Characteristics of hideout and hideout return can be detected from the outlet sodium chloride concentration profile when the heat flux of superheat* is applied to a crevice for a certain period of time to generate hideout. Figure 5 shows the hideout and hideout return result from the experiment [22].

* Superheat is heating (steam or other vapor not in contact with its own liquid) beyond its saturation point at a fixed pressure.

Figure 5. Experimental result by Rogers shows hideout and hideout return [22].

The studies of sodium hideout in SG crevices by Campan et al. [8], Finnigan et al. [10] and Millett [16] provided results about the effects of many controlling parameters.

Impurities concentration directly promotes the corrosion rates. In this study, sodium and sodium hydroxide were selected to study the effects on their concentration from several controlling parameters. Several important data were obtained from this set of experiments.

Campan compared the effect of concentration in the bulk water, high concentration of sodium at 50 ppm and low concentration at 5 ppm. As shown in Figure 6, higher concentration significantly assists the corrosion accumulation rate in the crevices.

Figure 6. Accumulation of sodium in the crevice during hideout phase, compared between 5 and 50 ppm of sodium in the bulk water [8].

Varying the primary loop's temperature has no significant effect on the accumulation of sodium concentration, while a huge effect on the accumulation is observed when varying the primary-secondary loop's temperature difference [8].

In this experiment, crevices were varied in size: the narrower the gaps, the more sodium concentration in the crevices. This fact indicates that using wider crevices might lessen the accumulation of impurities, which might mitigate the corrosion. However there is a limit to mechanical constraints in the SGs' system. In other words, if crevices are too wide, the tube support plates will not be able to support the tube bundle in an SG.

This experiment is a preliminary test to find the optimum condition to lower the Na concentration. In addition, Campan et al. suggested that further corrosion tests are required to study the correlation between the caustic concentration with IGA and IGSCC.

It is noted that even a small amount of sodium in the feedwater can causes severe corrosion problems as it accumulates through the water/steam cycle in the SG's operation [8].

Millett simulated the denting and fouling to study the transport processes in the PWR support crevices [16].

Millett set up another corrosion model for the PWR support crevice to measure the diffusion coefficient for chloride salt. The autoclave details are shown in Figure 7.

Figure 7. PWR support crevice experimental setup by Millett [16].

The measurement of effective diffusion coefficients indicates that a considerable diffusion resistance from the porous structure of non-protective magnetite exists, which is consistent with Campan's conclusion of pre-packed magnetite.

Finnigan et al. [10], Campan et al. [8] and Lumsden et al. [17] studied chemical concentration and corrosion in the PWR SG's tube sheet crevices. In Finnigan's study, an A508 carbon steel flat plate was welded to the column to create a crevice between the wall and the tube sheet as in the PWR SG. A layer of concentrated liquid contaminant was formed on the tube sheet.

There are many interesting results and data from the study by Finnigan et al. When the contaminant accumulates, it becomes higher in concentration and results in a higher boiling point, which makes these impurities harder to evaporate. The contaminant liquid tended to be formed at the crevice mouth but then built up at the crevice foot [10].

Although this effect is not significant, it was concluded that gravity could be a powerful driving force on the direct of corrosion. However, the surface tension at the tube can also lead to penetration into the horizontal direction and corrode the tube sheet. Evolution of nitrogen gas in the liquid can loosen the contaminant from the crevices.

In a special report by Strauss, metal oxide corrosion products (crud) were transported to the crevices and create more low flow areas at high-heat flux, which led to the propagation of corrosion [9]. Metal sludge pile-up on the tube sheet and the tube wall created more crevice-like conditions. Crud was formed by the effect of low pH from exhausted wet steam from a high-pressure turbine. Sludge build-up led to tube denting, IGA and IGSCC under its corroded environment.

Numerous models have been developed to describe mass and heat transfer processes inside the autoclave. For example, one of the models is used to describe a steady state process regarding correlation between the impurity concentrations in the bulk water and the gas phase [13].

$$C_k = C_b \exp[qS / \alpha \rho_w LD] \quad \text{Equation 1}$$

Where C_k and C_b are the concentration of evaporated and initial solution respectively,

q is the heat flux.

S is the length of the crevice or the thickness of the deposit.

ρ_w is the water density.

α is the porosity of deposits.

L is the heat of evaporation of water.

D is the diffusion coefficient of salt.

With a heat flux of 0.3 MW/m^2 (megawatt per meter square), the deposit thickness is 100 mm, the ratio of C_k/C_b is 105 while at the deposit thickness of 10 mm, and the C_k/C_b ratio is only 8. From this it can be implied that the higher the thickness, the more the impurities can evaporate from the initial solution and cause more severe attacks.

2.4 Dry Out Detection

In 1991, Balakrishnan [23] showed that crevice temperature profiles could be utilized to find steam blanketing. The existence of the steam blanketing phenomenon indicated that dry out was occurring. According to Balakrishnan's experiment, the increment of temperature profiles was detected by three thermocouples at different positions.

Balakrishnan's baseline experiment provided crevice temperature differences for normal operation.

The temperature profiles were detected by using thermocouples, which consisted of two different materials. The difference in electrical voltage between two metals was measured since voltage in metal varies at different temperatures.

Balakrishnan used three thermocouples to detect crevice temperature profiles. The superheat temperature increased with time for all thermocouples. The superheat temperature profile was raised from 50 °F to 66 °F for the middle thermocouple. When salt impurities were concentrated in the crevice, the saturation temperature (T_{sat}) was increased and the heat transfer coefficient (h) was also decreased in the steam blanket. According to Newton's law of cooling, as T_{sat} and h changes, the heat flux q also changes to maintain heat balance.

$$q = h(T_{sat} - T_s) \quad \text{Equation 2}$$

When dry out occurs, the steam blanket is formed. Since heat transfer coefficient in the steam is lower than in the bulk liquid phase, when the heat flux (q) is the same, the crevice temperature profile increases from the nucleate boiling phase at the beginning of the start up of the autoclave.

The steam blanket temperature distribution is provided in Figure 8.

Figure 8. Steam blanket temperature distribution in a carbon fiber-packed crevice [23].

According to Figure 8, when the crevice tube heater was on, the temperature profiles of the top and the bottom of the crevice gradually increased. The temperature profile of the middle of the crevice was expected to gradually increase similar to the top and the middle. However, after turning the crevice tube heater on for a while, the temperature increased sharply. This sharp increment of temperature indicated the existence of the steam blanket.

2.5 Measurement Techniques

Caustic (NaOH) was assigned to be the major cause of IGA and IGSCC in many research studies; for example, in Campan et al. [8], Finnigan et al. [10] and Mamet et al. [13], ^{24}Na as a radioactive tracer was selected to trace the concentration of the caustic in the SGs' corrosion models.

In a hideout study by Mamet et al. [13], ^{24}Na was measured at the blowdown sampling to find the decrement of sodium species in the bulk water with 5-7 $\mu\text{g/kg}$ (microgram per kilogram) of ^{24}Na , which is a typical range for the normal operation of PWRs.

The value of hideout (R) can be calculated from the ^{24}Na data:

$$R = \left(\frac{1 - \text{mass of Na removed with blowdown water}}{\text{mass of Na introduced in steam generator}} \right) \times 100\% \quad \text{Equation 3}$$

The study by Thompson focused on the measurement technique of corrosion deposition in the SG. The corrosion products that were removed from the SG can give us information about the chemical and physical characteristics of the deposits, such as concentration of chemical species, oxidation state of selected species and fouling mechanisms [12].

CHAPTER THREE

EXPERIMENTAL DESIGN AND PROCEDURES

3.1 Objectives

The objectives of this thesis are to prepare a high pressure and high temperature autoclave apparatus to study hideout and hideout return in the autoclave and verify the autoclave performance.

3.2 Experimental Methods

An existing autoclave that had previously been designed to simulate corrosion in the steam generator (SG) was used in this study. The autoclave can be used to evaluate the water chemistry and study corrosion mechanisms and other phenomena. The data obtained can be used to improve performance of real SGs in nuclear power plants.

A flow diagram of the experimental system used in these experiments is shown in Figure 9. The autoclave was fabricated by Rockwell International Inc. and was used in prior studies by Coonradt [1], Rogers [22] and Chou [24].

3.2.1 Feedwater System

The system piping is made of 1/4 -inch stainless steel 316 tubing with 0.35-inch wall thickness to handle the high pressure and temperature operating conditions. As shown in Figure 9, the process starts from a 30-gallon Nalgene feed tank, storing feedwater with the desired water chemistry inside. The feedwater consists of very pure DI water with a quality of approximately 1 megaohm mixed with sodium chloride at desired

concentration. Since the experiment used 60 ppm of NaCl, DI water of 1 megaohm is acceptable. However, if operation at lower ppm of NaCl is required, DI water with higher electrical resistance is recommended. The feedwater is pumped to the autoclave at the flow rate of 20 (ml/min) milliliter per minute through a Pulsar hydraulic diaphragm metering pump, which has a 5-45 ml/min flow range (SOP#2 in Appendix A^{*}). Since the pump has a large pulse characteristic, the pump required a dampener to keep the flow smooth. As shown in Figure 9, a pulse dampener was installed by putting a 25-foot length of 1/16- inch PEEK (Poly-ether-ether-ketone) tubing with a pressure rating of 204.14 atm^{**} (3000 psig^{***}) into the pump outlet. A check valve with a 136.1 atm (2000 psig) rating was then provided to direct the flow into a single direction. In case of power failure or pump failure, the check valve will prevent the system from flow reversal and hold pressure in the system. Two plug valves were installed as bypass valves at both the inlet and outlet of the autoclave. The plug valves' functions are for directing normal operation flow and bypassing the autoclave from the flow system when needed.

Since the heat from the autoclave itself is not adequate to elevate the temperature to the desired set point, a preheater was installed to preheat the feedwater before it enters the autoclave. A gravity convection oven "Blue M OV-12A" was used as a preheater (SOP#3 in Appendix A). First, the pressure is set to 27.22 atm (400 psig), then the autoclave bulk temperature is ramped from room temperature to 400 °F (204.4 °C) at the rate of 50 °F/hour. When the autoclave bulk temperature reaches 400 °F, the pressure is

^{*} Standard Operating Procedure#2 in Appendix A.

^{**} Atmospheric pressure.

^{***} Pound per square inch gauge.

increased to 60 atm (871 psig) and the temperature is then ramped up to the final autoclave bulk water temperature of 510 °F (265.55 °C). When the pressure is 60 atm and the temperature is 510 °F, the autoclave is run for at least 24 hours to let it reach steady state conditions.

Figure 9. Flow diagram of the experimental system [1].

3.2.2 Crevice

The simulated crevice between the tube support plate and the SG tubes (See Figure 1 in chapter one) was obtained by clamping a clamshell around the autoclave center tube as shown in Figure 10. The crevice tube heater simulated the heat source from the primary loop in a nuclear power plant SG. The bulk water of the autoclave simulated the bulk water in the secondary-loop in a nuclear power plant SG.

A cross section of the clamshell assembly is provided in Figure 10. As shown in Figure 10, the autoclave center tube containing a crevice tube heater was used to generate the heat to the crevice. The hideout phenomenon is expected to occur in the packed crevice when the crevice tube heater is turned on. The hideout return phenomenon is expected to occur after the hideout phase when the crevice tube heater is turned off.

The crevice tube heater contains a 600-watt cartridge heater with a diameter of 3 inches (7.62 cm^{*}.) long and 0.375 inch (0.953 cm.) wide. The heat transfer area of the heater is approximately 3.53 square inches (22.8 cm²). The crevice tube heater extends 0.9 inch (2.3 cm.) below the crevice to distribute uniform heat flux along the crevice. The bulk volume of the autoclave center tube is filled with eutectic salt (mixture of 54.3% KNO₃ by weight and 45.7% NaNO₃ by weight with the melting point of 482 °F ,250 °C), which is utilized as a heat transfer medium from the crevice tube heater's surface to the simulated crevice. Before turning on the crevice tube heater, the eutectic salt must be molten; otherwise, the crevice tube heater will break.

^{*} Centimeter.

Figure 10. Cross-section of the clamshell [22].

Figure 11. Top view and side view of half of a clamshell [24].

The clamshell is made from Inconel alloy 718. As shown in Figure 11, the clamshell consists of two main parts. These two parts can be attached together with screws. The advantage of this clamshell design is that when the two parts are separated, visual inspection can be performed without disturbing the interior contents in the crevice.

The clamshell is locked with two support rings by eight offset 316 stainless steel bolts. Both the top support ring and the bottom support ring are locked with two other attached rings. A Teflon doughnut is placed inside each attached ring. Under the normal high pressure and high temperature conditions of the autoclave operation, the Teflon doughnut swells and seals effectively to stop the fluid from entering the crevice created between each support ring and the autoclave center tube.

The gap between the clamshell and the autoclave center tube is 0.24 mm^{*} in diameter. The crevice length is 30.5 mm. The total crevice volume is approximately 447 mm³.

Thornel P-75S carbon fiber packing material was selected to accelerate the fouling process. The carbon fiber was originally rolled into a cylinder. The filament was rolled out and cut into pieces. Each piece of carbon fiber was placed on top of each other to create a crisscrossing pattern of 3 x 3 cm. mats (SOP#4 in Appendix A). A total 0.6562 grams of carbon fiber is filled in the crevice with a porosity of 30 percent^{**}. A carbon fiber mat with the total weight of 0.3281 gram was placed on each side of the clamshell and then clamped to the autoclave center tube Figure 12 shows where the carbon fiber mats are packed.

^{*} Millimeter.

^{**} See Appendix B for calculation.

Figure 12. Radial positions of each thermocouple [24].

In order to measure the superheat temperature profile, thermocouples were placed at different positions of the crevice. Radial positions of each thermocouple are shown in Figure 12. Temperature profiles in the crevice are expected to show a significant temperature change when dry out takes place.

As shown in Figure 13 the temperature profile of the middle of the crevice showed a sharp increment in the superheat phase, which indicates the presence of the dry out phenomenon [23].

Figure 13. Steam blanket temperature distribution in a carbon fiber-packed crevice [23].

3.2.3 Autoclave

The lid and the body of the autoclave are made from Inconel alloy 600 to represent the material of the tube in the real SG.

The feedwater exits the preheater and then enters the lower side of the autoclave via the inlet autoclave tube (SOP#5 in Appendix A). The water is further heated to the desired temperature, 510°F (265.55 °C) by the passive/active heaters around the autoclave (SOP#6 in Appendix A). The heated water is kept below the saturation point so it would remain in a liquid state at this temperature and a pressure of 60 atm (871 psig). The water leaves at the exit line located one inch below the top of the autoclave. The high-pressure outlet water with a temperature of about 510 °F (265.55 °C) then flows to a cooling water tank, where the exit flow is cooled down to about 75 °F (24 °C). For the hideout experiment, the crevice tube heater at the center of the crevice is turned on and

makes the crevice operate in the superheat condition (SOP#7 in Appendix A). The solution which was a little below saturation point is then evaporated around the crevice. The steam formed from the superheat condition exits the system. The crevice tube heater power was controlled by a variac*. Two multimeters were connected to the circuit to measure electric current and voltage. To avoid electrical shortage and overload, the variac was turned up at a rate of 50 watts/3 minutes.

The cooled water then flows through a pressure relief valve and a back pressure regulator valve (SOP#5 in Appendix A). The pressure relief valve was installed to release pressure at 82 atm (1,200 psig) in case of extremely high pressure to protect the system. The system's pressure is set to 60 atm (871 psig) by the back pressure regulator valve. The low-pressure exit flow is then directed to a series of measurement equipment before discharge to a waste drain.

* Voltage controller.

3.3 Measurement and Control of Parameters

Several measurement devices were installed to monitor and control parameters such as flow rate, temperature, and pressure. These devices need to be calibrated to ensure good control of experiments.

3.3.1 Temperature

According to Figure 9, thermocouples were installed at the autoclave inlet and cooling water outlet to monitor temperature. In Figure 10, two thermocouples temperature probes were installed at the core of the crevice tube heater. Eight probes near the crevice were installed in a spiral position as shown in Figure 12. These thermocouple probes were designed to measure the temperature profiles in the crevice. However, none of the eight thermocouples is currently functioning.

The center heater provides heat to the crevice to generate superheat. For safety reasons, fuses were installed to cut off the circuit and avoiding temperature runaways.

3.3.2 Pressure and Flow

The feedwater was pressurized to a high-pressure state through the Pulsar hydraulic diaphragm metering pump and a back pressure regulator to simulate a high-pressure condition in an SG. A pressure dampener was installed to keep the flow smooth. Two check valves were mounted to the flow system to avoid reverse flow, which could damage the injection pump. A pressure relief valve was installed after the cooling water apparatus to prevent pressure build up, which could result in an explosion.

3.3.3 pH

As per established molar ratio control (MCI) procedure [15, 18], the system's pH was maintained at the range of 5 –9. An online pH meter was used to monitor the pH of the solution. Since the pH meter has a certain operating temperature range, the samples must be cooled down before measuring the pH value.

3.3.4 Conductivity

Two conductivity meters with accuracies of ± 3 percent were installed to monitor the conductivity of the autoclave exit stream. Conductivity was used to detect hideout and hideout return phenomena. Conductivity was converted to the concentration of sodium chloride by using the calibration curves of known concentration of NaCl solutions [22]. The calibration curve generated is shown in Figure 17 in chapter five.

3.3.5 Data Collecting System

Temperature, pressure, pH, and conductivity measurement equipment were connected to a data acquisition system (DAQ). Different voltage signals were sent to the computer program "Lab View." Lab View is a control program, collecting data in a text file. The text file can be copied and transferred to a spreadsheet software and then can be analyzed.

3.4 Design of Experiment

After setting up the experimental system, one baseline experiment will be conducted to confirm that all equipment is fully functional. The baseline experiment data will also be used to find the magnitude of errors from the measurement. The baseline experiment will be run with the crevice without carbon fiber in order to find baseline hideout.

Theoretically, baseline hideout should be zero or close to zero. Then, three experimental runs will be performed to see the hideout and hideout return effect. Another two experimental runs without clamshell will also be performed to investigate experimental errors. All experiments will run with 60 ppm NaCl solution and 150 watts applied to the crevice for superheat. A summary of the design of the experiment is provided in Table 1.

Table 1. Experimental Matrix.

Run	Porosity
Baseline	100%
Packed-crevice #1	30%
Packed-crevice #2 confirmation	30%
Packed-crevice #3 confirmation	30%

3.5 The Experimental Setup

The experimental setup was based on parameters from the work of Coonradt [1] and Rogers [22] and Chou [24]. DI water was selected for this experiment. Carbon fiber was selected as a packing material with a porosity of 30 percent [22]. The steady state pressure and temperature were 510 °F (265.55 °C) and 60 atm (871 psig). Most of the experimental equipment was tested and calibrated before connecting to the experimental system.

3.6 Analysis of data

Hideout and hideout return phenomena are generally studied by analyzing the NaCl concentration profiles of the autoclave outlet. The disappearance of sodium chloride from outlet concentration indicates a hideout phenomenon. When the heat flux is removed from the crevice, a significant amount of sodium chloride is expected at the autoclave outlet as the result from the hideout return phenomenon.

According to mass balance, the area under the curve of hideout and the area under the curve of hideout return in Figure 14 should be about the same magnitude.

Conductivity profiles were measured and then converted to the sodium chloride concentration profiles, to analyze the amount of the NaCl hideout and hideout return.

Figure 14. Experimental result by Rogers shows hideout and hideout return [22].

After operating in the nucleate boiling region for a period of time, the steam blanket is expected to occur. If steam blanketing occurs anywhere in the crevice, the temperature at those positions will increase due to lower heat transfer coefficient of steam compared to boiling water. According to Balakrishnan's experiment, when there is no steam blanket, no noticeable sharp increment of a temperature profile is expected [23].

CHAPTER FOUR

RESULTS AND DISCUSSION

Prior to the experimental phase, the apparatus had to be set up and tested for repeatability and reproducibility. Measurement equipment was required to be calibrated.

4.1 Recommission and Verification

All equipment in the autoclave apparatus was checked and verified to ensure that it would work properly. Some equipment needed calibration for adjustment while some parts required total replacement.

4.1.1 Diaphragm Pump

At the beginning, the Pulsar hydraulic diaphragm metering pump was not able to pump. After the pump was disassembled and inspected, two balls in each ball valve were found instead of one. The extra balls were taken out. Later, the pump was reassembled and tested. After the pump worked properly, the flow rate of the pump was determined by measuring the amount of water coming out of the pressure regulator valve. To acquire a flow rate of 20 ml/min, the pump stroke was set at a 50 percent stroke rate.

4.1.2 Preheater

Since the original preheater was not operational, a new preheater along with the new tubing system for the preheater needed to be installed. A HP 5880A gas chromatography

oven chamber was modified to be a preheater. Its data integrator controlled the oven's temperature.

After using HP5880A gas chromatography (GC) as a preheater for a while, the oven's temperature could not be controlled. The investigation showed that the circuit board for temperature control in this GC was not functioning. A Blue M OV-12A gravity convectional oven was substituted for the broken preheater.

4.1.3 Measurement Equipment Exit Line Modification

As shown in Figure 15, the original measurement equipment exit line consisted of a number of 3-way and 4-way valves that were no longer needed. The old measurement equipment exit line was removed. The exit line was bypassed directly to the drain as shown in Figure 16.

Figure 15. Old measurement equipment exit line.

Figure 16. Modified measurement equipment exit line.

4.1.4 pH Meter

pH meter was calibrated and verified. The pH data are provided in Table 2.

Table 2. pH Meter Verification.

Standard	Reading
4.00	4.02
7.00	7.01

4.1.5 Precision Balance

A precision balance was used for weighing carbon fiber and optical grade NaCl. The balance was calibrated and its accuracy was verified by using a set of standard weights.

The verification data are provided in Table 3.

Table 3. Precision Balance Verification.

Standard Weight (g)	Reading (g)
20.0000	19.9999
10.0000	10.0000
5.0000	5.0001
2.0000	2.0001
1.0000	1.0019

Note: R^2 value for this calibration curve is 1. The precision balance is very accurate.

4.1.6 Conductivity Meter

Conductivity meters A and B were calibrated. Then a series of NaCl standard solutions of concentration 40, 60, 80, and 100 ppm were prepared and then measured by conductivity meters A and B. The data are provided in Table 4 and the resulting calibration curve is shown in Figure 17.

Table 4. Conductivity Meters Reading and Standard Solutions Correlation.

NaCl Standard (ppm)	Conductivity A $\mu\text{s/cm}$	Conductivity B $\mu\text{s/cm}$
40	85.85	84.1
60	126	128.3
80	173	153.4
100	209	208

Note: R^2 value for calibration curve of Conductivity A is 0.9942.

R^2 value for calibration curve of Conductivity B is 0.9814.

The relationship of NaCl standard solution and conductivity is linear as shown in Equation 4 and 5. Equations 4 and 5 were used to convert the measured conductivity into NaCl concentration in ppm.

Conductivity meter A:

$$y = 0.4791x - 1.1314 \quad \text{Equation 4}$$

Conductivity meter B:

$$y = 0.4949x - 0.9946 \quad \text{Equation 5}$$

Where

y = NaCl concentration (ppm).

x = Conductivity ($\mu\text{S}/\text{cm}$).

Figure 17. Conductivity calibration curve.

4.1.7 Thermocouples

The real time temperature reading of thermocouples, at the crevice tube heater #1 and #2, and the eutectic salt are close to the manual temperature measurement by a “Barnatt 155 thermocouple thermometer.” Tables 5,6, and 7 provide the verification data for the thermocouples measurement.

Table 5. Crevice Tube Heater #1 Temperature Reading.

Manual	154.3	204	253	301	347	394
DAQ	155	198.8	247.6	294.1	333.6	374.2

Note: $R^2 = 0.9993$.

Table 6. Crevice Tube Heater #2 Temperature Reading.

Manual	154.6	204	253	301	349	396
DAQ	156	205.3	254.1	305.9	353.1	399.7

Note: $R^2 = 0.9999$.

Table 7. Eutectic Salt Temperature Reading.

Manual	155	200	251	297	347	392
DAQ	150.7	195.2	243.4	281.5	323.5	366.4

Note: $R^2 = 0.9999$.

4.2 Experiment Results

Once the autoclave apparatus was recommissioned and all the measurement equipment were calibrated, the apparatus was ready to perform hideout and hideout return experiments.

Upon completion of the first run (run A) described in Table 8, the data showed too little hideout occurred, thus the original experimental matrix shown in Table 1 was modified to find out why the results were not consistent with previous studies in the same apparatus. Table 8 summarized all the experimental runs with descriptions and short notes. Each set of experiments will be described in detail in the following sections.

5.2.1 Three Experiments with 30 Percent Porosity

In the first experimental result, after the superheat of 150 watts from the crevice tube heater was applied to the crevice, a hideout phenomenon occurred immediately. First, the NaCl concentration dropped down and then climbed up. However, the NaCl concentration never reached its baseline value. When the NaCl concentration was steady and did not climb up anymore, the crevice tube heater was shut off and the hideout-return phenomenon occurred. At the hideout return phase, the NaCl concentration profile rose sharply and then dropped down to its original steady state value of 60 ppm. The hideout and hideout return for the first experiment are shown in Figure 18.

Table 8. Experimental Runs.

Run	Porosity	Clamshell	Crevice Tube Heater Power (Watts)	Hideout (mg)	Autoclave Inlet Temp °F	Note
A	30%	Yes	150	0	460	Wrong clamshell position.
1	30%	Yes	150	61.26	460	
2	30%	Yes	150	76.66	460	Confirmation run.
3	30%	Yes	150	62.98	460	Confirmation run.
B	100%	Yes	150	60.52	310	One Teflon doughnut was accidentally left out of the clamshell assembly.
4	100%	Yes	150	42.57	310	Baseline checked.
5	100%	Yes	150	42.09	310	Baseline checked.
C	NA*	No	150	54.37	310	Thick o-ring was used.
6	NA.	No	150	35.14	310	The eutectic salt level is above the crevice tube heater cartridge.
D	NA.	No	30	0	310	Varied amount of power.
E	NA.	No	50	0.49	310	
F	NA.	No	100	10.58	310	

Note: Experimental run A, B and C were run incorrectly so will not be discussed in the results and discussion section. However, the results of experiment A, B, C along with experiment D, E and F will be discussed in Appendix H.

Figure 18. Hideout and hideout return result.

* Not available.

Another experiment with the same conditions was conducted; this time pressure and temperature were more stable than the first experiment. However, the NaCl concentration profile did not reach its steady state value at 60 ppm as shown in Figure 19. The calculated hideout return value was too high which made the hideout efficiency* a little higher than unity. At this point it was suspected that the hideout process might occur somewhere else in the autoclave or another physical process account for the excess hideout value. However, the second experiment still showed the ability of the autoclave apparatus to produce the hideout and hideout return phenomena.

Figure 19. Hideout and hideout return confirmation experiment.

The last hideout and hideout return experiment result is similar to the result of the first experiment as shown in Figure 20. This indicated the ability of the apparatus to run and reproduce the experimental result. Data of three experiments for 30% packing porosity is shown in Table 9.

* See Appendix B for hideout efficiency calculation.

Figure 20. Hideout and hideout return from the other confirmation experiment.

Table 9. Summary of Total Accumulation for NaCl+DI Solution with 30% Packing Porosity and 150 Watts at the Superheat Phase.

Experiment	Hideout (mg)	Return (mg)	Hideout Efficiency	% Error
#1	61.26	63.81	0.814	
#2 (confirm)	76.66	72.19	1.027	26.167
#3 (confirm)	62.98	64.72	0.805	1.105

Note: Percent error based on #1 result.

4.2.2 Baseline Experiment

At the end of the third experiment, the preheater HP 5880A became dysfunctional. A Blue M OV-12A gravity convectional oven was installed. Since the new preheater's maximum operating temperature is 482 °F (250 °C), the operating ambient temperature in the preheater was set at 464 °F (240 °C) for the safety of the equipment. Even though the autoclave enter temperature was lower than the previous three experiments, the passive and active heaters attached to the autoclave had enough power to raise the autoclave bulk temperature to 510 °F (265.5 °C).

After three experimental runs, a baseline experiment was performed without packing material to find the error of the experiment. A clean concentric crevice without any packing material was attached to the autoclave. NaCl 60 ppm was pumped into the system and when the system was at a steady state in its operating condition of 60 atm (871 psig) and 510 °F (265.5 °C), 150 watts of power from the crevice tube heater was applied to the crevice.

Figure 21. Baseline experiment. No packing, 60 ppm NaCl and 150 watts.

Table 10. Baseline Total Accumulation NaCl 60 ppm and 0% Packed Crevice.

0% Packed Crevice	Hideout (mg)	Hideout Return (mg)	%Error
#4 (baseline)	42.57	48.97	1124.25
Rogers [22].	0	4	

Note: Percent Error based on Rogers [22].

According to Rogers' thesis [22], with the same experimental conditions*, only 4 mg of NaCl was detected in the hideout return phase. However, this baseline experiment's result contained about 40 mg of NaCl in the hideout and hideout return processes as shown in Table 10. This order of magnitude for this baseline hideout return is significant. Comparing the experiment results between Figure 21 and Figure 22, the baseline experiment did have more significant hideout than Rogers'. Further investigation was performed to find out what caused this large error.

Figure 22. Baseline experiment, 0% packed crevice and 150 watts by Rogers [22].

Obviously, some kind of physical process was occurring while the crevice tube heater was turned on. With almost the same amount of hideout and hideout return of NaCl, the possible physical processes were reduced to hideout/ hideout return and precipitation.

* Experiment conditions: NaCl 60 ppm solution, a clean concentric crevice, 150 watts applied to the crevice at the superheat phase.

4.3 Investigating Undesired Hideout Experiments

After finding a significant amount of NaCl disappearance and reappearing in the baseline experiment with a clean concentric crevice, the hideout was the first physical process that was suspected to account for this phenomenon.

Hideout phenomenon requires these conditions to occur:

- A little below saturation.
- Heat source to a packed crevice.
- Packed crevice.
- Solution with some concentration.

A series of experiments were performed to investigate the source of undesired hideout.

4.3.1 Old Surface-Corroded Clamshell as the Source of Hideout

It was suspected that the old surface-corroded clamshell was the source of the hideout. An experiment with the same condition of NaCl 60 ppm and 150 watts superheat was performed without the clamshell attached to the autoclave tube. The result of this experiment is shown in Figure 23.

Figure 23. Baseline experiment. No clamshell, 60 ppm NaCl and 150 watts.

According to the no-clamshell experiment result, significant hideout of 42.09 mg NaCl still occurred at the same level as when there was a clean concentric clamshell.

It can be concluded that the clamshell was not causing the undesired hideout phenomenon.

4.3.2 Autoclave Lid as the Source of Hideout

Hideout was still the prime concern in this investigation. There is a small gap found at the autoclave lid between the tube and the bottom of the autoclave lid. This gap was suspected of being a crevice. The eutectic salt level was high enough to reach that gap.

In order to verify this, the autoclave lid was put in the oven at a high enough temperature to melt the eutectic salt (higher than 482 °F, 250 °C). Some eutectic salt was poured out. This resulted in the eutectic salt level being only a little higher than the top position of the crevice tube heater cartridge as shown in Figure 10.

Another experiment with the same condition of NaCl 60 ppm solution and 150 watts applied to the crevice for superheating was performed without the clamshell attached to the autoclave center tube. The result of this experiment is shown in Figure 24.

Figure 24. Baseline experiment result: no-clamshell, 60 ppm NaCl and 150 watts when the eutectic salt level above the crevice tube heater cartridge.

Table 11. Baseline Total Accumulation NaCl 60 ppm. Decrease Eutectic Salt Level.

0% Packed Crevice	Hideout (mg)	Hideout Return (mg)	%Error	Note
No Clamshell	35.14	28.19	604.75	The eutectic salt level is above the crevice tube heater cartridge.
Rogers [22].	0	4		%Error based on Rogers [22].

A significant amount of hideout still occurred at the same level when there was a clean concentric clamshell as shown in Table 11. It can be concluded that the eutectic salt level was not causing the undesired hideout phenomenon.

All of the experimental data is summarized in table 12.

Table 12. Summary of Experimental Results.

Run#	Percent Porosity	Clamshell	Hideout (mg)	Hideout Return (mg)	Note
1	30	Yes	61.26	63.81	
2(confirm)	30	Yes	76.66	72.19	
3(confirm)	30	Yes	62.98	64.72	
4(baseline)	100	Yes	42.57	48.97	
5	NA.	No	42.09	35.62	
6	NA.	No	35.14	28.19	The eutectic salt level is above the crevice tube heater cartridge.
Rogers [22].	100	Yes	0	4	Reference baseline data.

4.3.3 Precipitation as a Cause of NaCl Disappearance and Reappearance

In every single experimental run, when the crevice tube heater was turned on, there always was a pressure fluctuation of 20 psi or more. This indicated that too much boiling was occurring in the autoclave. This boiling phenomenon might have caused NaCl to precipitate, and the precipitated NaCl stayed dry in the autoclave. Once the crevice tube heater was turned off, the pressure fluctuation was gone. Then the precipitated NaCl

re-dissolved back into the bulk water and returned after the crevice tube heater was turned off.

An experiment to find the relationship between pressure and temperature was conducted. As shown in Figure 25, the pressure was ramped up to 150 psig (9.2 atm) and the temperature was ramped up to 300 °F (148.89 °C). When the system reached a steady state, the pressure was ramped down and when the pressure reached 100 psig (5.8 atm), the pressure started to fluctuate between 90 - 100 psig (5.12-5.8 atm). This pressure fluctuation indicated that the autoclave bulk water was boiling. However, according to the steam table at 300 °F (148.89 °C) the saturation point is approximately 66 psig (3.49 atm).

Figure 25. Pressure and temperature correlation.

Since the steam table is accurate, the cause of the problem was determined be the pressure measurement accuracy.

After checking with the manufacturer of the pressure gauge, the “zeroing pressure gauge” step had been omitted. This step was not done in any experiment for this thesis study. Zeroing the pressure gauge can be done by adjusting the screw at the “Z” position. The reading of pressure by the pressure gauge is lower than the real pressure by 20 psi.

An experiment to verify the pressure gauge was set up. A full nitrogen tank equipped with a pressure gauge and a pressure regulator was used as a pressure source and pressure control. The nitrogen tank pressure was 2,000 psig (135.09 atm). A tubing system was connected starting at the nitrogen tank going through the pressure transducer that was connected to a digital pressure gauge.

Table 13. Pressure Test Experiment.

Nitrogen Tank Pressure Gauge (Psig)	Pressure Transducer (Psig)	Digital Pressure Gauge (Psig)
202	215	200
300	316.8	297
400	419.34	398
500	514.34	495
600	616.45	598
700	719.25	699

According to the experimental result of the pressure test as shown in Table 13, the pressure reading from the digital pressure gauge agreed with the nitrogen tank pressure gauge. It was concluded that after zeroing the digital pressure gauge, the digital pressure

gauge was reading the pressure accurately. There was about 20-psi differences between the pressure transducer gauge and digital pressure gauge reading, which could be easily adjusted by changing the equation for the pressure transducer at the data acquisition (DAQ).

4.3.4 Low Flow as a Cause of NaCl Disappearance and Reappearance

The Pulsar hydraulic diaphragm metering pump flow capacity was decreased after the pump was running for several months. After checking the cause of low flow from the pump's manual, the check valve of the pump was replaced. Some flow was regained; however, the flow was still lower than the pump flow capacity specifications.

In conclusion, to avoid precipitation in the experiment, a higher flow rate is required. Running an experiment at higher pressure could also reduce the precipitation problem caused by pressure fluctuation into the saturation region. The back pressure regulator also required at least a 15 ml/min flow rate in order to maintain constant back pressure in the system.

CHAPTER FIVE

FUTURE WORK

Certain equipment needs to be fixed as follows:

Table 14. Future Maintenance.

Diaphragm Pump	Replace bypass valve seat.
	Replace piston seal.
Preheater Coil	Replace preheater coil.
Back Pressure Regulator	Replace internal parts.

Once the diaphragm pump is fixed, the preheater coil is replaced and the back pressure regulator is fixed, a simple hideout experiment with the condition of high pressure of 890 psig (60.56 atm) and autoclave bulk temperature of 510 °F (265.55 °C) should be performed. The flow of the feedwater should be at least 20 ml/min. Those changes are expected to reduce pressure fluctuation in the system. When the pressure fluctuations are reduced, precipitation caused by saturation conditions will be reduced. After the precipitation problem is eliminated, this experiment should result in a minimum amount of NaCl disappearance and reappearance. If the expected results occur, then the autoclave performance will be confirmed and packed crevice experiments can be continued.

CHAPTER SIX

CONCLUSIONS

The diaphragm metering pump, dispensing pump, preheater, autoclave, passive and active heaters, crevice tube heater, pH meter, conductivity meters, thermocouples, and DAQ system were successfully recommissioned, tested, and verified. Only a few modifications are required to complete recommissioning of the experimental system.

The packed-crevice experiment results did prove the ability of the autoclave system to reproduce the experiment result within a 5 percent error range.

The investigation showed that the undesired disappearance and reappearance of NaCl is most likely caused by precipitation. A simple test to reduce precipitation of NaCl is proposed in Chapter Five.

Once the problem of precipitation of NaCl is eliminated, the study of hideout and hideout return and verification of performance of the autoclave can be continued.

REFERENCES

1. R.S. Coonradt, "*PAA Studies in a Simulated Steam Generator Crevice*," Master's Thesis San Jose State University, (1999).
2. J. Douglas, "*Solutions for Steam Generators*," EPRI Journal, 29-34 (May/June 1995).
3. Energy Information Administration, *Electric Power Monthly*, (January 2004).
4. P.J. Millett, "*Transport Processes in Steam Generator Crevices. I. General Corrosion Mode*," Corrosion Science, **41**, 2165-2190 (1999).
5. P.J. Millett, "*Transport Processes in Steam Generator Crevices II. A Simplified Method for Estimating Impurity Accumulation Rates*," Corrosion Science, **41**, 2191-2210 (1999).
6. O.S. Tatone, P. Meindl and G.F. Taylor, "*Steam Generator Tube Performance: Experience with Water-Cooled Nuclear Power Reactors During 1983 and 1984*," Nuclear Safety, **28** (3), 374-390 (July-September 1987).
7. U.S. Nuclear Regulatory Commission, "*Nuclear Reactors*," [Online] Available at <http://www.nrc.gov/reading-rm/basic-ref/students/reactors.html>, (accessed February 11, 2004).
8. J.L. Campan and C.E. Shoemaker, "*Sodium Hideout Studies in Steam Generator Crevices*," Environmental Degradation of Materials in Nuclear Power Systems-Water Reactors Edited by G.J. Theus and J.R. Weeks, the Metallurgical Society, 199- 207 (1988).
9. S.D. Strauss, "*Special Report Water Treatment*," Power, 15-32 (May 1994).
10. D.J. Finnigan and J.E. Forrestm, "*Chemical Concentration and Corrosion in PWR Steam Generator Tubesheet Crevices, Water Chemistry of Nuclear Reactor System 5, Volume 1*," (International conference organized by British Nuclear Energy Society held in Bournemouth on 23-27 October 1989).
11. B. Nair, "*Steam Gnerator Replacement and Repair Technology Continues to Advance*," Power Engineering, **23**, 23-27 (April 1997).
12. R.H. Thompson, "*Deposits Gage Effectiveness of Plant Water-Chemistry Programs*," Power, edited by S. Strauss, 11-16 (March 1989).

13. V.A. Mamet and O.I. Martynova, "Hideout in NPP Steam-Generator Boiler Water and Its Effect on the Operating Reliability of Equipment," *Thermal Engineering*, 40 (7), 497-502 (1993).
14. N.S. McIntyre, R.D. Davidson, T.L. Walzak, A.M. Brennenstuhl, F. Gonzalez and S. Corazza, "The Corrosion of Steam Generator Surfaces Under Typical Secondary Coolant Conditions: Effects of pH Excursions on The Alloy Surface Composition," *Corrosion Science*, 37 (7), 1059-1083 (1995).
15. P.J. Millett, "Steam Generator Degradation: Current Mitigation Strategies for Controlling Corrosion," (Presented at CNRA/CSNI Workshop on Steam Generator Tube Integrity in Nuclear Power Plants, October 30-November 2, 1995).
16. P.J. Millett, "Fundamental Study of Support Plate Crevice Transport Processes in PWR Steam Generators," Master's Thesis University of Connecticut, (1898).
17. J.B. Lumsden, G.A. Pollock, P.J. Stocker and P.J. Millett, "Hideout of Na in a Prototypic SG Tube/Tube Support Plate Heated Crevices," EPRI RPS407-13, Electrical Power Research Institute: Palo Alto, California, (July 1997).
18. R. Riess and P.J. Millett, "State of the Art in Nuclear Plant Cycle Chemistry," (Presented at the 12th International Conference on the Properties of Water and Steam, Orlando, Florida, September 11 – 16, 1994).
19. G.M.W. Mann and R. Castle, "Salt Concentration in Heated Crevices and Simulated Scale," NP-3050 Research Project 1171-3 Topical Report, EPRI, (October 1983).
20. P.J. Millett and J.M. Fenton, "A Modeling Study of Parameters Controlling Local Concentration Processes in Pressurized Water Reactor Steam Generators," *Nuclear Technology*, 108, 256-265 (November 1994).
21. W.E. Berry, "Corrosion Standards and Control in Nuclear Power Industry, Chapter 8," *Industrial Corrosion Standards and Control* by I.H. Cocks, (1973).
22. P.M. Rogers, "Sodium Chloride Hideout in a Simulated Steam Generator and Tube Support Plate Crevice," Master's Thesis San Jose State University, (1997).
23. P. Balakrishnan, "Hideout and Return of Complex Mixtures in Crevices," EPRI S407-13, (September 1991).
24. D. Chou, "The Effect of PAA on NaCl Hideout in a Simulated Steam Generator Crevice," Master's Thesis San Jose State University, (2000).

APPENDIX A

STANDARD OPERATING PROCEDURES

Standard Operating Procedure #1 (SOP#1)

Feedwater Tank

A cylindrical, 15-gallon tank with cover and spigot, made of High Density Poly Ethylene (HDPE).

Warning

1. Eliminating oxygen is crucial to avoid corrosion in the system.
2. Be careful not to put any contaminants into the feedwater tank.

Start Up

1. Transfer DI water from the DI water sources to a 15-gallon Nalgene feedwater tank as shown in Figure A1.

Note: DI water sources are in the lab E203 and lab E311.

Nitrogen Gas
Inlet Line

Figure A1. Feedwater tank.

2. Use the programmable dispensing pump model DP200 to pump 15 gallons of DI water from the Nalgene tank to the feedwater tank (See SOP#9).
3. Once the feedwater tank is filled with DI water, turn on the nitrogen gas by turning on the pressure regulator knob connected to the nitrogen tank. The feedwater tank diagram is shown in Figure A2.
4. The nitrogen gas line is connected to the top of the feedwater tank cover. (See Figure A1). Hold the tank cover; use the end of nitrogen gas line to fill the air space above the water surface.
5. Put the tank cover on top of the feedwater tank to prevent air leakage into the tank and to feed nitrogen gas to get rid of dissolved oxygen (DO) in the feedwater. Adjust the pressure regulator knob to control the amount of nitrogen gas bubbling into the feedwater tank. The amount of gas bubbling should be enough to cause mixing in the feedwater tank.
6. Once the level of the oxygen is zero, lower the nitrogen intake, but keep measuring the DO concentration to make sure that the oxygen level in the feedwater stays low.
7. Check water quality, make sure that
 - Feedwater pH is 5 – 6.
 - Feedwater Conductivity is “near zero.”
 - Feedwater Dissolved Oxygen (DO) concentration is “below zero.”
8. Write down baseline information about DI water: conductivity, pH, dissolved oxygen and temperature.
9. Start pumping DI water into the system by turning on the Pulsar hydraulic diaphragm metering pump (See SOP#2).

Operation

1. Measure the DO concentration in the feed tank periodically to make sure that the level of the oxygen is still zero.
2. Fill the tank with DI water when necessary.

Shut Down

1. Turn off the Pulsar hydraulic diaphragm metering pump (See SOP#2).
2. Shut off nitrogen feed valve.
3. Release the pressure on the nitrogen regulator diaphragm.

Figure A2. Feedwater tank diagram.

Troubleshooting

- If any leak is detected especially at the fitting on top of feedwater tank, it can cause low flow.

Switch the bypass valves to bypass the feedwater from the autoclave to maintain high pressure in the autoclave. The experimental system diagram is shown in Figure A3. Stop the Pulsar hydraulic diaphragm metering pump and fix the leak. Once the leak is fixed, switch the bypass valves back to normal position and restart the Pulsar hydraulic diaphragm metering pump.

Figure A3. Experimental system diagram.

Standard Operating Procedure #2

Pulsar Hydraulic Diaphragm Metering Pump

A diaphragm pump that can handle pressure up to 1,000 psig.

A variac connected to the pump to provide power.

Warning

1. The system is running at high pressure; make sure you wear goggles at all times.
2. The PEEK flexible tubing should be secured, so that it will remain secured if it splits under pressure.

Figure A4. Pulsar hydraulic diaphragm metering pump.

Start Up

1. Make sure that the eccentric box shown in Figure A4 is filled with the Pulsalube 7H (purple) hydraulic oil.
2. Inspect the level of the hydraulic oil. Add oil if the oil level is lower than the spring coil.
3. If water or condensation is present in the eccentric box, change the oil.
4. Set stroke percent by turning the adjustable stroke knob shown in Figure A4.

5. Start the pump by turning on the variac's toggle switch on to 0 – 120 volt.

Note: There is also a 0-140 volt setting.

6. Set the variac dial to the desired percent (usually 100 percent).
7. Calibrate the flow versus the stroke percent by measuring the volume of water flowing through the tubing right after the back pressure regulator (unscrew the fitting to measure the flow) per a period of time at each stroke percent.

Operation

1. Set the stroke percent to the setting required to achieve the desired flow rate.
2. Inspect the pump to make sure that there is no leakage at the discharge. If any leak is present, stop the pump and tighten the nut or replace the nut.

Optional:

Occasionally, verify the actual flow rate with the stroke percent by measuring the volume of water flowing through the tubing right after the back pressure regulator (unscrew the fitting to measure the flow) per a period of time at several stroke percentages and confirm they fit the calibration curve.

Shut Down

- Short term

1. Turn the variac toggle switch to "Off."

- Long term (more than 1 month)

1. Turn the variac toggle switch to "Off."
2. Turn the variac percent dial down to zero.
3. Reduce the stroke percent to zero by turning the adjustable stroke knob.

Troubleshooting

- If the pump is running but there is little or no flow, it is likely that:
 1. The diaphragm might be broken. Disassemble the diaphragm and replace with a new one.

2. There are two balls in the check valve exit. Each check valve should have one ball seated at the exit of the check valve to direct the flow. Two balls will stop the flow. Remove one extra ball.
 3. The check valve is installed in the wrong direction.
- If the flow gradually drops, it is likely that:
 1. The check valve may be leaking. Clean the check valve or replace if necessary.
 2. The suction line may be leaking. Find and fix the leak.
 3. The ball valves may be worn. Replace the ball valves.

Note: Exploded view for each component of the Pulsar hydraulic diaphragm metering pump is shown in Figure A5.

Figure A5. Pulsar hydraulic diaphragm metering pump exploded view.

More information about the Pulsar hydraulic diaphragm metering pump can be found at "Pulsar hydraulic diaphragm metering pumps," Bulletin No. PMP-IOM-96, Pulsarfeeder, A unit of IDEX Corporation.

Standard Operating Procedure #3

Preheater Blue M OV-12A

An electrical gravity-convection oven that can heat up to 500 °F (260 °C).

Warning

1. The preheater operates at a high temperature range, be careful not to touch the body of the preheater during operation.
2. Do not disassemble the tubing system while operating since a water leak may generate electric shock.
3. Do not operate above 482°F, (250 °C); the preheater could be damaged.

Note: This preheater temperature unit is in centigrade.

There are three ranges of power selection on each temperature range as follows:

Low: 100.4 – 170.6 °F (38-77 °C).
Medium: 172.4-325.4 °F (78 - 163 °C).
High: 372.2 – 500 °F (164-260 °C).

Figure A6. Preheater Blue M OV-12A.

Start Up

1. Plug the preheater to a 120-volt power source (wall).
2. Turn the “Power Selector Dial” as shown in Figure A6 from “Off” to “Low, Medium or High” position as desired. Select the correct power setting for each temperature set point.

Note: The “Red, Main Power Light” should be on.

3. Turn the “Temperature Control Dial” shown in Figure A6 to the desired temperature set point.
4. In the normal start up operation, slowly ramp up temperature by 50 °F/hour (27.8 °C/hour) until the desired operating condition is reached.

Operation

1. Frequently check the preheater’s ambient temperature by connecting the “Baenant 155 thermocouple thermometer” to the thermocouple on the top of the preheater to make sure that the operating condition for the water in the preheater is no above the saturation point.
2. Always keep the water system pressure above the saturation point by adjusting the back pressure regulator appropriately (See SOP#5).

Note: The yellow light on the panel indicates that the preheater is adjusting the temperature to the set point.

Shut Down

1. Slowly turn the “Temperature Control Dial” to room temperature along with the “Power Selector Dial” to match each temperature range. Eventually, the “Power Selector Dial” should be at the “Off” position.

Troubleshooting

- If temperature does not increase:
 1. Check if the preheater is connected to the power source or not.
 2. Check the fuse. If the fuse is broken, replace it.

Standard Operating Procedure #4

Clamshell assembly

Clamshell assembled on the autoclave tube to form a crevice in the autoclave.

Warning

1. DO NOT touch carbon fiber directly. Wear gloves.
2. Use appropriate facemask to avoid inhalation of carbon fiber.
3. Always wear goggles.

Start Up

1. Calculate the amount of THORNEL[®] carbon fiber required.

For example, for 30 percent porosity;

$$\text{Porosity \%} = \frac{(\text{Total crevice volume} - \text{Carbon fiber volume})}{\text{Total crevice volume}} \quad \text{Equation 6}$$

Total crevice volume

$$30 = (0.447 - V_{\text{carbon}}) \times 100 / 0.447$$

$$V_{\text{carbon}} = 0.3129 \text{ ml.}$$

Carbon fiber's density is 2.1 g/ml, therefore, $0.3129 \text{ ml} \times 2.1 \text{ g/ml.} = 0.6571 \text{ gram}$ of carbon fiber is required.

Put $0.6571 / 2 = 0.3129 \text{ gram}$ of the carbon fiber on each side of the clamshell.

Figure A7. Mat formation from carbon fiber.

2. Cut the carbon fiber from the carbon fiber roll into 4 cm. sections long and lay them in a crisscross pattern, layer by layer to form a mat with an irregular channel as shown in Figure A7.

Figure A8. Lay each mat on each side of the clamshell.

3. Cut the carbon-fiber mats into two 3 x 3 cm. squares.
4. Weigh carbon-fiber mats, add or reduce amount of carbon fiber to get 0.3129 gram on each mat.
5. Put one mat on each side of the clamshell as shown in Figure A8.
6. Check every single clamshell thread and screw. If a screw cannot go in to the thread, go to a workshop, rethread with the taper lubricated with oil.
7. Put the Teflon doughnuts into the crevice supports, two on each side, top and bottom as shown in Figure A9.
8. Assemble the top crevice support and slide it into the autoclave center tube.

9. Bring the two parts of the clamshell with the prepared carbon fiber of the desired porosity and put them on the autoclave tube where the bottom of the crevice must be exactly 9 cm. from the tube ending to ensure that the crevice is aligned with the tube heater and the thermocouples as shown in Figure A9. Secure the crevice by the nuts.
10. Use the screws to connect the top crevice support to the clamshell then assemble the bottom crevice support and connect it to the clamshell (See Figure A9).
11. Secure the position of the clamshell by putting an Inconel o-ring at the bottom of the clamshell (See Figure A9).

Operation

NA.

Shut Down

1. Once the autoclave lid is disassembled (See SOP#5), use the appropriate Allen wrench to disassemble each part of the clamshell.
2. Start by removing the Inconel o-ring at the bottom of the clamshell.
3. Slowly slide the clamshell out of the autoclave center tube.
4. Use the Allen wrench to disassemble the bottom and top support rings.
5. Use the Allen wrench to disassemble the support rings.
6. Remove the Teflon doughnuts from the support rings.
7. Use the Allen wrench to disassemble the clamshell.

Troubleshooting

- If you cannot screw or unscrew the nuts, use WD-40 spray to lubricate the thread.
- If the screw is stuck, ask a lab assistant for assistance.

E

Figure A9. Cross-section of clamshell assembly [22].

Standard Operating Procedure #5

Autoclave

A pressure vessel designed to operate at high temperature and pressure.

Warning

1. The autoclave operates at a high pressure of 871 psig and high temperature. Make sure to follow the standard operating procedure to avoid accident/damage.
2. Do not lift the autoclave with your hands as the welding can be irreversibly damaged.
3. DO NOT raise the autoclave temperature above saturation point at any time since steam will be generated.

Start Up

1. Fill the autoclave column with about 500-600 ml. of deionized (DI) water to the top of the autoclave.
2. Put a new carbon fiber o-ring onto the autoclave.

Note: A new carbon fiber o-ring can be prepared by using the old carbon fiber o-ring as a guide on the new John Crane carbon-fiber sheet. Use a pen to mark the outer and inner circle of the o-ring. Use scissors to cut out the fiber sheet to form an o-ring as shown in Figure A10.

Figure A10. John Crane carbon fiber o-ring.

3. Assemble the autoclave by holding the autoclave tightly with a large special wrench provided by Rockwell shown in Figure A11. Slowly tighten each pair of bolts that are opposite to each other, pair by pair to make the autoclave level. Tighten each bolt twice; a little torque is applied each time.
4. Put the fiberglass heat insulation around the autoclave.

Figure A11. Assemble the autoclave lid to the autoclave.

Operation

1. Connect thermocouples and other measurement equipment to the data acquisition (DAQ) (See SOP#8).
2. Start logging experimental data on Labview. The data should be saved on C:\SJSU\ACHRIVE\sample.txt (See SOP#8).
3. Start the process by turning on the Pulsar hydraulic diaphragm metering pump at the desired percent stroke to fill the water in the process. Inspect if there is any leakage in the process (See SOP#2).
4. Turn on the digital pressure gauge by pressing the "PSI" button. Zero the digital pressure gauge by using a small screwdriver to turn the "z" screw until the panel shows "000."
5. Set the system pressure to the desired pressure by cranking up the back pressure regulator knob. The system pressure is monitored by a digital pressure gauge and analog pressure gauge shown in Figure A12.

Figure A12. Experimental system diagram.

6. Check if the passive and active heaters are connected to the power sources. If not plug them into the power sources.
7. Turn on the variac labeled "Active." Turn the knob to 68 percent to regulate the appropriate amount of power put into the passive and active heaters.
8. Ramp up the temperature of the preheater, passive heater and active heater at the rate of 50 °F/hour (27.8 °C /hour) manually by using the temperature controller until the desired temperature is reached. Slowly ramp up to avoid temperature overshooting since it can cause system instability.

Allow 24 hours of operation; analyze data to make sure that steady state is reached.

9. When the system reaches a steady state, switch to desired test solution. Desired test solution example is NaCl 60 ppm.
10. Allow 6 hours of residence time and 1-2 more hours for steady state. Write down baseline information.
11. Observe the exit line frequently to monitor flow rate.
12. Observe pressure to see if any pressure fluctuation exists.

Shut Down

1. Turn off preheater.

2. Set passive heater and active heater temperature to 32 °F.
3. Switch to DI water and flush the system.
4. Remove fiberglass heat insulation.
5. When the bulk temperature reaches room temperature, reduce pressure by cranking down the back pressure regulator. When the pressure is lower than 300 psig, turn off the pump.
6. Disconnect thermocouples to the data acquisition (DAQ).
7. Turn off the computer.
8. Lower the active variac knob to 0 percent. Switch off variac switch. Turn off passive and active heater control by disconnecting the power supply.
9. Stop the Pulsar hydraulic diaphragm metering pump (See SOP#2).
10. Use the wrench to secure the autoclave and unscrew the 12 bolts.
11. Remove autoclave lid. Place it on the metal support.
12. Use a masterflex pump to remove bulk water out of autoclave.
13. Disassemble the clamshell (See SOP#4).

Troubleshooting

- If leakage is detected:
 1. Shut down preheater, passive and active heaters.
 2. Remove the fiberglass heat insulation. Let the system run overnight until room temperature is reached.
 3. Stop the Pulsar hydraulic diaphragm metering pump (See SOP#2).
 4. Use the wrench to secure the autoclave and unscrew the 12 bolts.
 5. Remove autoclave lid. Place it on the metal support.
 6. Use a masterflex pump to remove bulk water out of autoclave.

7. Disassemble the clamshell (See SOP#4).
8. Replace the leaking tube with a new tube and fittings connected to the tube.
9. If leakage is found in the tube connected to the autoclave, the tube needs to be replaced by cutting out the old tube and connecting the new high pressure stainless steel 316, (SS316) tube onto the autoclave by welding.

Note: Welding SS316 to Inconel alloy 600 require a special welding rod
"Inconel 82."

Standard Operating Procedure #6

Active Heater and Passive Heater

Two dual band “HOT WATT” heaters wrapped around autoclave to control autoclave temperature.

Warning

1. High heat and high electric current are involved with this equipment. Make sure to follow the standard operating procedure to avoid accident/damage.

Figure A13. Active and passive heater temperature controller.

Start Up

1. Connect the Fuji Electric active and passive temperature control boxes as shown in Figure A13 to a power source.
2. The display will show the default of set value “SV” of 32 °F and PV of current autoclave bulk temperature.
3. Press “SV” button to set the set point temperature.
4. Use arrow key “^” or “v” to select desired temperature and then press “ENT.”

Operation

1. Observe bulk temperature from the DAQ and do some manual temperature reading by using a “Barnatt 155 thermocouple thermometer” or equivalent to make sure that the passive and active heaters are working properly.

Shut Down

1. Set the active and passive heaters temperature to 32 °F.
2. Let the system run overnight.
3. The active and passive temperature should reach room temperature. Lower the active variac to 0 percent, switch off. Turn off the passive and active heater control boxes by disconnecting the power supply.

Troubleshooting

NA.

Standard Operating Procedure #7

Crevice Tube Heater

A cartridge heater immersed in the eutectic salt solution. (Refer to Figure A9 as the crevice tube heater).

Warning

1. Do not turn the crevice tube heater on if the eutectic salt temperature is less than 482 °F (250 °C) since the eutectic salt is not molten and thus, will not transferring heat. The power given to the crevice tube heater will be turned into heat and destroy crevice tube heater wires.
2. Turning the crevice tube heater variac knob too fast will cause electric spike, which can break the tube heater.
3. Make sure that the eutectic salt level is at least above the crevice tube heater.

Start Up

1. Use a "Barnatt 155 thermocouple thermometer" or equivalent to measure the eutectic salt temperature. Make sure the eutectic salt temperature is higher than 482 °F (250 °C). Also measure the autoclave bulk temperature.

Note: Eutectic salt is the mixture of KNO_3 54.3 wt%* and NaNO_3 45.7 wt%.
The eutectic salt melting point is 482 °F (250 °C).

2. Use a multimeter to detect any shortage on the crevice tube heater. The resistance should be as follows:
 - Between each side of the crevice tube heater plug: 100 ohms.
 - Between each side of the crevice tube heater plug and metal casing: Overload.
3. Connect power cord from the crevice tube heater to the power outlet labeled "TUBE."
4. Connect voltmeter and amp-meter to the crevice tube heater circuit.
(See Figure D3 in Appendix D).

* Percent by weight.

5. Switch the toggle switch of the variac, labeled "TUBE" from "Off" position to 0-120 volt.
6. Set the Fuji Electric temperature controller labeled "TUBE" to 1000 °F.
7. Slowly turn the TUBE variac knob while monitoring voltage and ampere at the rate of 50 watts per 3 minutes until the desired power is reached.

Note: Power can be calculated from $P \text{ (watts)} = I \text{ (ampere)} \times V \text{ (volt)}$.

Operation

1. Monitor the temperature of the crevice tube heater 1 and 2, the eutectic salt, the autoclave enter and the autoclave exit.
2. Monitor data extracted from the LabView program.

Shut Down

1. Set the Fuji Electric temperature controller labeled "TUBE" to 32 °F.
2. Slowly turn down the TUBE variac to 0 percent.
3. Switch the TUBE variac toggle switch to zero.
4. Disconnect power cord from TUBE outlet box and the crevice tube heater.

Troubleshooting

- If the voltage cannot be ramped up:
 1. Fuse is broken; check TUBE variac's fuse with multimeter. If the fuse is broken, replace with a new fuse with the rating of 10 or 12-ampere and 250 volts.
 2. Heater is broken, use a multimeter to determine resistance;
 - Between each side of the crevice tube heater plug:

If the resistance is very high, the crevice heater is broken.

 - Between each side of the crevice tube heater plug and the crevice tube heater metal casing:

If the resistance is very low, there is a shortage between the crevice tube heater wire and its own metal casing.

- Eutectic Salt level too high or too low:
 1. If the eutectic salt level is too high above the crevice tube heater, heat from crevice tube heater can be transferred to somewhere else other than crevice and might cause undesired steam generation.
 2. If the eutectic salt level is lower than the crevice tube heater, heat will not be able to transfer to the entire crevice and cause error in the hideout and hideout return experiment.

Note: Make sure that the eutectic level is a little above the top position of the cartridge crevice tube heater.

Standard Operating Procedure #8

Data Acquisition (DAQ) and Measurement Equipment.

All measurement equipment is connected to the data logging system. While the experiment is running, the data logging system will record each parameter data.

Warning

1. Be careful not to let the DAQ wires contact any heat source directly.

Start Up

1. Connect all thermocouples with the DAQ connectors as shown in Figure A11.

Figure A11. Thermocouple and a DAQ connector.

2. Connect the pH meter controller, conductivity meters controllers and pressure transducer to the DAQ connectors.
3. Connect pH meter probe to the tubing system. Make sure there is no leakage.
4. Turn on the conductivity meters controllers "A" and "B" model "Cole Parmer 19101-00." The switch is located at the back of conductivity meters.

5. Turn on the "HP 6177C DC Current Source" by turning the switch from "off" to "amp."
6. Turn on the computer.
7. Click start button, select "My recent documents."
8. Select these files:
 - Archive
 - Master
 - Process
 - Display

The windows of these files will be opened.

Operation

1. At the archive window as shown in Figure A12, designate path and name of the data file C:\SJSU\ACHRIVE\111504_rampup.txt. Set the desired default archive rate. The default archive rate is setting of how frequent the data will be logged. The default archive rate is normally set at 0.5 to 1 min.

Figure A12. Archive window.

2. Set computer time to the current time.
3. At the Master window, click “right arrow” button as shown in Figure A13 to start logging data.

Figure A13. Master window.

4. The real time experimental data will be collected.
5. Periodically back up the logged data in text format (.txt) on another storage device.

Shut Down

1. At the Master window, click the “red circle” button to stop logging data.
2. Close down the four windows.
3. Turn off the computer.
4. Disconnect all thermocouples from the DAQ connectors.
5. Disconnect the pH meter probe from the tubing system and put the pH sensor probe to the pH 4 solution to preserve the probe.
6. Turn off the conductivity meters “A” and “B.”

Note: DAQ setup for all equipment is shown in Appendix F.

Troubleshooting

- If a thermocouple do not read temperature:

Check if the wire inside the DAQ connector is broken or not. If the wire is broken, reconnect the wire.

Standard Operating Procedure #9

Dispensing Pump DP200

A dispensing pump used for pumping accurate amount of DI water.

Warning

NA.

Figure A14. Dispensing pump DP200.

Start Up

1. Turn on the dispensing pump by pressing “Power” as shown in Figure A14.
2. Press “Manual Remote” to remove all air/bubbles in the suction and discharge line.

3. Calibrate the dispensing pump by following these steps;
4. Put the tube ending of the pump discharge line into a dry 50 ml volume cylinder.
5. Press "Volume Output", read the amount of water in the cylinder.
6. Punch in the value of volume and press "Volume Input."
7. Test the accuracy by removing all air/bubbles and setting volume to 50 ml and cycle to 2. Put the suction hose in a dry 100 ml volume cylinder. Then press "Auto Contin." If the calibration is accurate, the amount of water in the cylinder should be 100 ml. If not, start over from 4.

Operation

1. When the calibration is accurate, set desired volume and cycle of pumping to match your desired amount of DI water to be transferred.
2. Press "Clear" and then press "Auto Contin."

Shut Down

1. Turn off the Dispensing Pump by pressing "Power."

Troubleshooting

NA.

APPENDIX B

START UP AND SHUTDOWN PROCEDURE FOR THE SIMULATED STEAM GENERATOR

General Precautions:

1. This apparatus is running at high pressure and high temperature, with a lot of high electrical voltage and current involved. Be careful and be alert at all times when running this apparatus.
2. Every single component of this apparatus is important. They are there for a reason. DO NOT TAMPER WITH or REMOVE ANY PART of APPARATUS WITHOUT FULL KNOWLEGDE of WHAT YOU ARE DOING. The whole system can be damaged with such misconduct.
3. Eutectic Salt will not transfer heat unless it is molten at 482 °F (250 °C) or above.

Safety Checks

1. Test the power supply to make sure that all switches, lights and controls are functioning properly.
2. Inspect all piping connections and tanks to see if there is any leak.

Caution: Make sure of this at the startup of the process.

3. Make sure there are no bubbles in the exit line by keeping the water below the saturation point.
4. Do not harm the weak points of the autoclave such as joints.
5. System pressure must never exceed 1,200 psig.

Total Startup Procedure for the Experimental System

1. Prepare the desired packing material and put it in the crevice.
 2. Fill the autoclave column with deionized (DI) water (about 500-600 ml to make sure that DI water is filled throughout the autoclave).
 3. Assemble the autoclave.
 - 3.1 Assemble the clamshell (See SOP#4 in Appendix A).
 - Check every single clamshell thread by turning the screw. If a screw cannot go in to the thread, go to a workshop, rethread with the taper lubricated with oil.
 - Use a measuring tape to locate the position of the crevice. The bottom of the crevice must be exactly 9 cm. from the tube end to ensure that the crevice is aligned with the tube heater and the thermocouples.
 - 3.2 Put a new carbon fiber o-ring onto the autoclave.
 - 3.3 Assemble the autoclave by holding the autoclave tightly by a large special wrench provided by Rockwell. Tighten each pair of bolts in the opposite direction, pair by pair to make the autoclave level. Tie every bolt securely.
- Note: Special attention is needed when screwing and unscrewing since the inlet and outlet tubes of the autoclave are weak and cannot be welded again.
4. Connect thermocouples to the DAQ (See SOP#8 in Appendix A).
 5. Make sure all valves are at the correct position for the process. Check the process line. Make sure they are correct and the tubing connections are securely fastened.
 6. Check water quality, make sure that
 - Feedwater pH is 5 – 6.
 - Feedwater Conductivity is “near zero.”
 7. Write down baseline information about DI water: conductivity, pH and temperature.
 8. Fill the DI water into the feed tank.
 9. Turn on the inert N₂ gas.

10. Zero the pressure gauge.
11. Turn on cooling ball valve, not the on/off valve. Keep the water flow just enough to cool the coil (preventing system from overshooting).
12. Inspect the hydraulic fluid for the diaphragm pump. Make sure that the oil level is above the spring. Set the feedwater flow rate to 20 ml/min.
13. Start logging experimental data on Labview. The data should be saved on C:\SJSU\ACHRIVE\....txt.
14. Turn on the Pulsar hydraulic diaphragm metering pump to fill the water in the process. Inspect if there is any leakage in the process.

Note: Pressure cannot build up if any leak is present.

15. Set the system pressure to 400 psig (26.21 atm) by turning the back pressure regulator valve (Read the pressure using a digital pressure gauge and an analog pressure gauge).
 16. Turn on variac for active heater and turn the knob to 68 percent power.
 17. Ramp up the temperature of preheater, passive heater and active heater at the rate of 50 °F/hour manually until the temperature reaches 400 °F (204.45 °C). Slowly ramp up to avoid overshooting and damaging the system (See SOP#6 in Appendix A). The temperature ramping sequence is shown in Table B1 and Table B2.
- Check if the passive/active heaters are connected to the power sources. If not, connect them to the power source.

Table B1. Temperature ramp up time sequence (at 26.21 atm, 400 psig).

Hour	Preheater Temperature °C	Passive Heater Temperature °F	Active Heater Temperature °F
	24	67	70
0:00	58	120	120
0:30	72		
1:00	85	170	170
1:30	99		
2:00	113	220	220
2:30	127		
3:00	141	270	270
3:30	154		
4:00	168	320	320
4:30	182		
5:00	196	370	370
5:30	204	400	400

18. When the autoclave bulk temperature is 400 °F (204.4 °C) set pressure to 871 psig (60 atm).
19. Slowly ramp up the temperature until the system reaches a steady state.

Table B2. Temperature ramp up time sequence (at 60 atm, 871 psig).

Hour	Preheater Temperature °C	Passive Heater Temperature °F	Active Heater Temperature °F
6:00	210	420	420
7:00	225	435	435
8:00	230	450	450
9:00	235	475	475
10:00	235	490	490
11:00	235	510	510

Allow 24 hours of operation; analyze data to see if steady state is reached.

Steady State is recognized by;

- Bulk temperature : 510 °F (256.55 °C).
- Pressure : 60 atm (871 psig).
- pH : 5 – 6.
- Salt concentration calculated from total conductivity should be ~ 0 ppm.
- Analyze data by putting in graphical form.

Write down baseline information.

Note: It takes about 12 hours from the beginning to reach the operating conditions.

20. When the system reaches a steady state, switch to NaCl solution feedwater tank.

21. Allow 6 hours of resident time and 1-2 more hours for steady state. Write down baseline information.

Caution: The eutectic salt must be melted to ensure proper heat transfer before turning on the crevice tube heater.

22. Manually measure and record the temperature of the crevice tube heater and the bulk solution by using thermometer before turning ON the crevice tube heater.

23. Turn on crevice tube heater (See SOP#7 in Appendix A).

23.1 First, check the crevice tube heater variac fuse to see if it is broken or not.
If the fuse is broken, replace with a new fuse with the rating of 10 or 12-ampere and 250 volts.

23.2 Check the resistance before connecting the crevice tube heater to its power outlet.

- Resistance between two heater lines should be about 100 ohms.
- Resistance between each heater line and its casing ideally should be at overload or very high (megaohm level).

- 23.3 Use a power extension to connect the crevice tube heater to its power supply outlet.
- 23.4 Set the crevice tube heater's temperature to 1000 °F.
- 23.5 Switch on the variac to 0 - 120 volt range. (Push the switch backward).
- 23.6 Slowly turn on the crevice tube heater's variac at the rate of 50 watts/3 minutes. Set power to 150 watts.
- Note: Measure voltage and electrical current while turning the variac to control the wattage. The wattage can be calculated by $P = IV$ (1 watt = 1 ampere x 1 volt).
27. The conductivity will drop immediately; wait until the conductivity reaches the baseline value.
28. Manually measure and record the temperature of the crevice tube heater and the autoclave bulk solution by using a thermometer before turning OFF the crevice tube heater.
29. Turn off the crevice tube heater.
30. Analyze data and see if hideout return is acquired.
31. Switch to DI water and flush the system until it reaches the DI baseline values.

Shutdown Procedure for the Experimental System

1. Turn off the crevice tube heater and turn off the preheater.
2. Set the passive and active heaters temperature to 32 °F.
3. Switch to DI water and flush the system.
4. When the autoclave bulk temperature reaches room temperature, reduce pressure. When the system pressure is lower than 300 psig, turn off the Pulsar hydraulic diaphragm metering pump.
5. Lower the active variac to 0 percent, switch off. Turn off passive and active heater controllers.

APPENDIX C

MISCELLANEOUS PROCEDURES

Preparing 10 gram of Eutectic Salt

1. Weigh 5.4300 grams of KNO_3 (Potassium nitrate) using a precision balance.
2. Weigh 4.5700 grams of NaNO_3 (Sodium nitrate) using a precision balance.
3. Pour both KNO_3 and NaNO_3 into a mortar.
4. Crush both kinds of salt together with a pestle.

Putting Eutectic Salt into the Autoclave Center Tube

1. Put the mortar containing the crushed eutectic salt mixture into an oven.
2. Heat the oven up to 260 °C (500 °F). When the oven's temperature reaches 500 °F, leave the eutectic salt for 10 minutes. The salt should now be molten.
3. Use a pair of heat resistance gloves to handle the hot mortar.
4. Carefully and quickly pour the molten eutectic salt into the autoclave tube.

Note: Liquid eutectic salt rapidly hardens into a solid at room temperature.

Preparing 50 Liters of NaCl 60 ppm solution

1. Weigh 3.0000 grams of NaCl (Sodium chloride) 99% grade into a beaker using a precision balance.
2. Use a dispensing pump to transfer 50 liters of DI water into the Nalgene tank, which is located under the rotating motor (See SOP#9 in Appendix A).
3. Turn on the dispensing pump by pressing "Power."

4. Press "Manual Remote" to remove all air/bubbles in the suction and the discharge line.
5. Calibrate the dispensing pump by using a dry 50 ml volume cylinder.
6. Press "Volume Output," read the amount of water in the cylinder.
7. Punch in the value of volume and press "Volume Input."
8. Test the accuracy by removing all air/bubbles and set volume to 50 ml and cycle to 2; put the suction hose in a dry 100 ml volume cylinder. Then press "Auto Contin." If the calibration is accurate, the amount of water in the cylinder should be 100 ml. If not, start over from 4.
9. When the calibration is accurate set volume to 80 ml and cycle to 625. Then press "Clear" and then press "Auto Contin."
10. Wait for about 1-2 hours for the pump to finish pumping 50 liters of DI water into the Nalgene tank.
11. Install the mixer to the rotating motor above the Nalgene tank using an Allen wrench. Connect the motor controller to the power source.
12. Turn on the mixer.
13. Use some DI water in the Nalgene tank to dissolve NaCl then pour the solution into the mixing tank. Repeat this step until all solid NaCl is dissolved and all NaCl in the beaker is transferred to the Nalgene tank.
14. Leave the mixer on for about 2 hours; measure the conductivity as a baseline.

Calculation of Hideout Efficiency

Hideout 61.26 mg.

Hideout Return 63.80 mg.

Hideout time 556 minutes.

Observed accumulation (hideout) rate:

61.26 mg/556 minute = 0.110 mg/minute.

Calculated accumulation (hideout) rate:

$$\frac{0.4 \times \text{Power (watts)} \times \text{NaCl conc. (ppm)}}{\Delta H_{\text{vap}} (\text{kJ/Kg})} \times \frac{1 \text{ L}}{1 \text{ Kg}} \times \frac{3.6 \text{ kJ}}{1 \text{ Watt-hour}} \times \frac{1 \text{ hour}}{1 \text{ minute}} \quad \text{Equation 7}$$

$$0.4 \times 150 \text{ Watts} \times 58.8 \text{ mg/L} \times 1\text{L}/1\text{Kg} \times 3.6 \text{ kJ}/1 \text{ Watt-hour} = 0.135 \text{ mg/min}$$

$$1564.1 \text{ kJ/Kg} \times 60 \text{ minute/hour.}$$

$$= 0.135 \text{ mg/minute.}$$

$$\text{Accumulation (hideout) Efficiency} = 0.110/0.135 = 0.814.$$

Note: conc. is concentration.

Measure the Voltage and the Electrical Current of the Crevice Tube Heater

The measurement can only be performed when the eutectic salt is molten at 482 °F (250 °C).

Select the proper range of the voltage to 0-200 volt and the current to 2 amperes.

1. Connect the voltmeter to the outlet. Read the voltage.
2. Connect the amp-meter to the outlet. Switch the line to amp-meter to let the electric current flow directly into the amp-meter. Read the current.

Carbon Fiber Preparation

General instruction

1. Use hand gloves. Avoid touching the carbon fiber directly.
2. Use air mask to avoid inhalation.

Procedure

1. Calculate the amount of carbon fiber needed; for example, 30 percent porosity;

$$\text{Porosity\%} = \frac{(\text{Total crevice volume} - \text{Carbon fiber volume})}{\text{Total crevice volume}} \quad \text{Equation 8}$$

$$30 = (0.447 - V_{\text{carbon}}) \times 100 / 0.447$$

$$V_{\text{carbon}} = 0.3129 \text{ ml.}$$

$$\begin{aligned} \text{Carbon fiber's density is } 2.1 \text{ g/ml, therefore, we need } 0.3129 \text{ ml} \times 2.1 \text{ g/ml} \\ = 0.6571 \text{ gram.} \end{aligned}$$

$$\text{Put } 0.6571 / 2 = 0.3285 \text{ gram of the carbon fiber on each side of the crevice.}$$

2. Cut the carbon fiber about 4 cm. long and lay them in a crisscross pattern, layer by layer.

3. Cut the carbon fiber mats into two 3 x 3 cm. squares.
4. Weigh carbon fiber mats, add or reduce amount of carbon fiber to get 0.3285 gram on each mat.
5. Put each mat on each crevice.

Crevice Tube Heater Removal Procedure

Before proceeding, make absolutely sure that you wear heat-resistant gloves and safety glasses.

Procedure

1. First of all, make sure that the new crevice tube heater is functioning properly by measuring the resistance to make sure that there is no short circuit.

The resistances should be as follows:

- Between two lines: 100 ohms.
- Between each line and the casing: Overload.

2. Wear heat-resistant gloves and safety glasses.
3. Use a temperature reader to read the eutectic salt temperature.

Note: The eutectic salt melts at 482 °F (250 °C) or above.

4. Unscrew the power cord.
5. Unscrew the four bolts at the top of the plate.
6. Gently remove the crevice tube heater. Since the crevice tube heater is bundled with thermocouples, these thermocouples will come off along with the crevice tube heater.

Note: Avoid damaging crevice thermocouples soldered to the wall.

7. Remove the thermocouples that are attached to the crevice tube heater while the eutectic salt is still molten.

Note: Be very careful since it is very hot at 482 °F (250 °C), it can easily burn you.

8. Remove the heater casing while it is still hot and put it into a new crevice tube heater.
9. Cover the autoclave's middle hole with heat insulation. Otherwise, it will lose some heat and cause the salt to get sticky and it will be hard to put the crevice tube heater back in.
10. Attach thermocouples for the crevice tube heater 1, the crevice tube heater 2 and the eutectic salt. Bind them to the crevice tube heater with two wires, one at the bottom and one at the top of the crevice tube heater.
11. Connect the plate holder to the crevice tube heater and measure the length to the heater casing.
12. Connect the crevice tube heater wires to the power cord.
13. Check the temperature of the eutectic salt; it should be at 482 °F (250 °C) or above.
14. Slowly put the bundle of the crevice tube heater and thermocouples into the inner side of the autoclave center tube, make sure not to push it too hard or too fast. When the cold crevice tube heater contact the molten eutectic salt, the salt will transfer some heat to the crevice tube heater and make the eutectic salt itself sticky. The key is to wait for a while until the salt is heated up so that it is molten again and then you can slowly continue to put the crevice tube heater into the eutectic salt.

Note: It is very important not to break the crevice thermocouples inside the autoclave center tube.

Thermocouples Wires Stripping

1. Use an appropriate size of wire stripper for each wire.
2. To strip the wire, put the wire into the guide plate hole. Once the wire reaches the die, apply some pressure and then turn the wire stripper.
3. When the stripping is started, keep turning the wire stripper to peel the wire. This action is similar to sharpening a pencil.

Note: After each cut and turn, remove all stripped wire since it might cause fouling and hinder the stripping. If anything is stuck in the guide hole, use a screwdriver to remove the guide plate and clean the hole. Do not reversely turn the wire stripper since it might break the point of the die or break the wire.

APPENDIX D ELECTRICAL DIAGRAM OF HEATERS

Figure D1. Electrical power control diagram of the active heater for the autoclave.

Figure D2. Electrical power control diagram of the passive heater for the autoclave.

Figure D3. Electrical power control diagram of the crevice tube heater for the autoclave.

APPENDIX E

TROUBLESHOOTING

Table E1. Troubleshooting.

Equipment	Problems	Possible causes	Solution
Pulsar series diaphragm pump	No outlet flow.	Diaphragm leaks.	Replace with a new diaphragm.
		Check valves installed in the wrong direction.	Disassemble and reinstall following the instruction manual.
		Two balls in the check valve.	Remove one ball from the check valve.
Autoclave	Cannot build up pressure.	Broken carbon fiber o-ring.	Replace with a new carbon fiber o-ring.
		Leakage in tubing or fittings.	Find the leakage and replace tubing, tighten the fittings.
	Pressure fluctuates too much.	Steam is generated.	Lower operating temperature.
	Passive/active heaters do not respond.		Reset by disconnecting power source and reconnect.
	Do not get proper reading of the crevice tube heater's current and voltage.	Crevice tube heater's electrical shortage.	Check ohm between two lines (should be ~ 100 ohms), between line and its casing (should be off-line). If shortage is found (~ 0 ohm), replace tube heater.
	Pressure drop.	Loose Fittings.	Shutdown all heaters, cool down and tighten fittings.
		Leakage.	Find the leak and try to seal/fix it.

Clamshell	Cannot unscrew bolt.	Corrosion	Spray rust loosener.
	Cannot screw bolt.	Bad thread.	Rethread with a taper.
Thermocouple	Cannot read properly.	Thermocouple is broken.	Replace with a new thermocouple.
		Wiring problems.	Check the wiring of the thermocouples and DAQ. Rewire, rethread the thermocouple wire.
Cooling water	Low Flow.	Corrosion in fittings.	Unscrew fittings and remove corroded products.

APPENDIX F **DATA ACQUISITION DIAGRAMS**

Figure F1. DAQ display.

Figure F2. Archive window.

Figure F3. Master window.

Figure F4. Process window.

Figure F5. DAQ diagram.

Figure F6. Conductivity A setting.

Figure F7. System pressure setting.

Figure F8. Conductivity B setting.

Figure F9. pH meter setting.

Figure F11. Autoclave inlet thermocouple setting.

Figure F13. Autoclave exit thermocouple setting.

APPENDIX G

CHEMICALS AND EQUIPMENT

Chemicals

- N₂ gas
- Eutectic salt mixture (weight percent: 54.3%KNO₃, 45.7%NaNO₃)
- Conductivity standard NaCl
- 1-megaohm deionized water (DI water)
- Sodium chloride optical grade 99.99%
- Inert carbon fiber

Equipment

- Nalgene tanks (Cole-Parmer: 3-15 gallons, 1-10 gallons)
- Plastic ¼" feed tubing
- Dispensing pump (Masterflex)
- Hydraulic diaphragm metering pump (Pulsar-Feeder: 30ml/min, 1000 psig)
- Poly-Ether-Ether-Ketone tubing (length=50 ft, ID=0.25 mm, OD=1.6 mm)
- Blue M OV-12A gravity convection oven
- 1-Liter high pressure alloy 718 autoclave
- 8 K type thermocouples (Omega)
- Electronics pressure sensor (Cole Parmer)
- Pressure gauges (PSItronics)
- pH cell and meter (Corning)
- Total specific conductance cell and meter (Cole-Parmer)
- Back pressure regulator (GoReg BP-60)
- Pressure relief valve
- On/Off valves (Sunnyvale valve: SS-42S4)
- Switching valves (Sunnyvale valve: SS-42XS4)
- Check valves
- ¼" 316 Stainless steel tubing
- Pentium 133 MHz data acquisition system
- Temperature controllers
- Tube fittings, elbows, T's
- Mixer and motor
- Heater bands (Omega)
- Trash can heat exchanger
- Autoclave and tubing insulation
- Clamshell assembly
- Pressure fittings

APPENDIX H

ADDITIONAL EXPERIMENTAL RESULTS

Experiment A

This was the first experiment conducted after the experimental system was recommissioned. The system pressure fluctuated more than 50 psig. The packed crevice was oriented correctly around the crevice tube heater, so that no hideout and hideout return phenomena were observed in this experiment.

Figure G1. The first experiment with clamshell placed on the wrong position.

Experiment B

In this experiment one Teflon doughnut was accidentally left out of the clamshell assembly. This created an undesired crevice between the clamshell and the autoclave center tube and resulted in higher values of hideout and hideout return.

Table G1. Hideout and Hideout Return Results for No Clamshell Experiment and Experiment B.

0% Packed Crevice	Hideout (mg)	Hideout Return (mg)	Note
No Clamshell	42.54	48.97	
Experiment B	60.52	60.75	One Teflon doughnut was accidentally left out of the clamshell assembly.

Experiment C

In this experiment a thick carbon fiber o-ring for the autoclave lid was used instead of a normal thin carbon fiber o-ring. However, no significant difference in the amount of hideout and hideout return was found between the experiments with the thin o-ring and the thick o-ring.

Table G2. Hideout and Hideout Return Results for No Clamshell Experiment and Experiment C.

0% Packed Crevice	Hideout (mg)	Hideout Return (mg)	Note
No Clamshell	42.54	48.97	Thin o-ring.
Experiment C	54.37	48.15	Thick o-ring.

Experiment D, E and F

Experiment D, E and F were performed to investigate the effect of the crevice tube heater power towards the amount of the hideout occurred. The experiment results are summarized in Table G3.

Table G3. Baseline Experiment Result When the Crevice Tube Heater Powers were Varied.

Run	Porosity	Clamshell	Crevice Tube Heater Power (watts)	Hideout (mg)	Autoclave Inlet Temp °F	Note
D	NA.	No	30	0	310	
E	NA.	No	50	0.49	310	
F	NA.	No	100	10.58	310	

The higher the power applied from the crevice tube heater to the autoclave bulk NaCl solution, the higher amount of hideout was obtained. This set of experiment result is consistent with the precipitation theory, the more power applied, the more precipitation is likely to occur. However, the experiment must be conducted at 150 watts power, so this set of experiment data was not used to compare with the experiment data in chapter five.