

REPORT DOCUMENTATION PAGE			Form Approved OMB NO. 0704-0188		
<p>The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA, 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.</p>					
1. REPORT DATE (DD-MM-YYYY) 24-09-2014		2. REPORT TYPE Final Report		3. DATES COVERED (From - To) 10-May-2011 - 9-Jul-2014	
4. TITLE AND SUBTITLE Final Report: PWAS EMIS-ECIS Active Carbon Filter Residual Life Estimation Methodology			5a. CONTRACT NUMBER W911NF-11-1-0210		
			5b. GRANT NUMBER		
			5c. PROGRAM ELEMENT NUMBER 206023		
6. AUTHORS VICTOR GIURGIUTIU, JINGJING BAO			5d. PROJECT NUMBER		
			5e. TASK NUMBER		
			5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAMES AND ADDRESSES University of South Carolina Research Foun 901 Sumter ST, 5th floor Byrnes  Columbia, SC 29201 -3961			8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211			10. SPONSOR/MONITOR'S ACRONYM(S) ARO		
			11. SPONSOR/MONITOR'S REPORT NUMBER(S) 60011-CH.1		
12. DISTRIBUTION AVAILABILITY STATEMENT Approved for Public Release; Distribution Unlimited					
13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.					
14. ABSTRACT The impregnated active carbon used in air purification systems degrades over time due to exposure to contamination and mechanical effects (packing, settling, flow channeling, etc.). Existing methods for the estimation of the residual life of the carbon filter has difficulties differentiating the impedance changes due to chemical contamination from those due to mechanical changes. In this project a novel approach is proposed to use piezoelectric wafer active sensors (PWAS) embedded into the carbon bed to evaluate the overall chemical and mechanical conditions of the carbon bed during SO <sub>2</sub> contamination tests. On one hand, the electromechanical					
15. SUBJECT TERMS active carbon filter, residual life estimation, impedance spectroscopy, piezoelectric active wafer sensor, PWAS, electromechanical impedance spectroscopy, EMIS, electrochemical impedance spectroscopy, ECIS					
16. SECURITY CLASSIFICATION OF:		17. LIMITATION OF ABSTRACT		15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU	UU		Victor Giurgituiu
					19b. TELEPHONE NUMBER 803-777-8018

## Report Title

Final Report: PWAS EMIS-ECIS Active Carbon Filter Residual Life Estimation Methodology

### ABSTRACT

The impregnated active carbon used in air purification systems degrades over time due to exposure to contamination and mechanical effects (packing, settling, flow channeling, etc.). Existing methods for the estimation of the residual life of the carbon filter has difficulties differentiating the impedance changes due to chemical contamination from those due to mechanical changes. In this project a novel approach is proposed to use piezoelectric wafer active sensors (PWAS) embedded into the carbon bed to evaluate the overall chemical and mechanical conditions of the carbon bed during SO<sub>2</sub> contamination tests. On one hand, the electromechanical impedance spectroscopy (EMIS) of the PWAS indicates the mechanical impedance changes of the carbon bed; on the other hand, the ECIS of the PWAS indicates the conductivity change of the carbon bed due to the formation of sulfuric acid during the SO<sub>2</sub> contamination. An impedance model was created to simulate the PWAS-carbon under increasing pressure. A carbon bed contamination apparatus is designed and constructed to perform the SO<sub>2</sub> contamination experiments. A data analysis method was developed to extract the small variation of the PWAS EMIS peak. An US patent (US 8,814,996 B2) was awarded during the project.

---

**Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:**

**(a) Papers published in peer-reviewed journals (N/A for none)**

<u>Received</u>	<u>Paper</u>
-----------------	--------------

**TOTAL:**

**Number of Papers published in peer-reviewed journals:**

---

**(b) Papers published in non-peer-reviewed journals (N/A for none)**

<u>Received</u>	<u>Paper</u>
-----------------	--------------

**TOTAL:**

**Number of Papers published in non peer-reviewed journals:**

---

**(c) Presentations**

Number of Presentations: 0.00

---

**Non Peer-Reviewed Conference Proceeding publications (other than abstracts):**

Received      Paper

**TOTAL:**

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

---

**Peer-Reviewed Conference Proceeding publications (other than abstracts):**

Received      Paper

**TOTAL:**

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

---

**(d) Manuscripts**

Received      Paper

**TOTAL:**

Number of Manuscripts:

---

**Books**

Received      Book

**TOTAL:**

Received

Book Chapter

**TOTAL:**

---

**Patents Submitted**

---

**Patents Awarded**

US 8,814,996 B2 "Methods and Sensors for the Detection of Active Carbon Filters Degradation with EMIS-ECIS PWAS"  
~~awarded 2014/08/26~~

---

**Awards**

---

---

**Graduate Students**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
ERIK FRANKFORTER	0.20	
TUNCAY KAMAS	0.80	
BANIBRATA PODDAR	0.40	
<b>FTE Equivalent:</b>	<b>1.40</b>	
<b>Total Number:</b>	<b>3</b>	

---

**Names of Post Doctorates**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	
JINGJING BAO	0.75	
<b>FTE Equivalent:</b>	<b>0.75</b>	
<b>Total Number:</b>	<b>1</b>	

---

**Names of Faculty Supported**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
VICTOR GIURGIUTIU	0.00	No
<b>FTE Equivalent:</b>	<b>0.00</b>	
<b>Total Number:</b>	<b>1</b>	

---

### Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
JAMES GOLD	1.00	Mechanical Engineering
MIRANDA DUNCAN	1.00	BIOMEDICAL ENGINEERING
ASHLEY VALOVICIN	1.00	BIOMEDICAL ENGINEERING
<b>FTE Equivalent:</b>	<b>3.00</b>	
<b>Total Number:</b>	<b>3</b>	

### Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: ..... 3.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 3.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 3.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense ..... 1.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:..... 0.00

---

### Names of Personnel receiving masters degrees

<u>NAME</u>
<b>Total Number:</b>

---

### Names of personnel receiving PHDs

<u>NAME</u>
<b>Total Number:</b>

---

### Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
<b>FTE Equivalent:</b>	
<b>Total Number:</b>	

---

### Sub Contractors (DD882)

## Inventions (DD882)

### 5 Methods and Sensors for the Detection of Active Carbon Filters Degradation with EMIS-ECIS PWAS

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) Y

Foreign Countries of application (5g-2):

5a: VICTOR GIURGIUTIU

5f-1a: UNIVERSITY OF SOUTH CAROLINA

5f-c: 300 MAIN ST

COLUMBIA SC 29208

5a: GLENN RUBEL

5f-1a: ECBC, APG ABERDEEN PROVING GROUNDS, US

5f-c: 2 Halls Cross Roads

ABERDEEN MD 21001

5a: GREGORY WILLIAM PETERSON

5f-1a: ECBC, APG ABERDEEN PROVING GROUNDS, US

5f-c: 2 Halls Cross Roads

ABERDEEN MD 21001

5a: JINGJING BAO

5f-1a: UNIVERSITY OF SOUTH CAROLINA

5f-c: 300 MAIN ST

COLUMBIA SC 29208

### Scientific Progress

1. Developed a PWAS-carbon bed impedance model, mechanical and chemical degradations that are relevant to carbon filter residual life estimation will be considered
2. Developed specialized experiments to validate and verify the model
3. Developed data analysis methods and residual life estimation methodology
4. Designed and constructed a bench top demonstrator to test the overall methodology
5. Specified the requirement for a proof of concept prototype development and write up the final report

### Technology Transfer

VISITED THE ECBC ARMY RESEARCHERS IN 2013 AND MADE A COMPREHENSIVE PRESENTATION OF THE RESEARCH RESULTS AND MAIN FINDINGS

INTERACTED WITH THE ECBC ARMY RESEARCHERS ON A WEEKLY BASIS THROUGH EMAIL AND/OR PHONE TO REPORT PROGRESS AND GET INPUT

SUBMITTED MONTHLY AND QUARTERLY PROGRESS REPORTS TO ECBC ARMY RESEARCHERS AND IMPLEMENTED THEIR COMMENTS AND SUGGESTIONS IN OUR RESEARCH PROCEDURES

**FINAL PROGRESS REPORT**

**PWAS EMIS-ECIS Active Carbon Filter  
Residual Life Estimation Methodology**

Submitted by:

Victor Giurgiutiu and Jingjing Bao  
University of South Carolina  
300 Main Street, Columbia SC 29208  
Telephone: (803) 777-8018  
Email: [victorg@sc.edu](mailto:victorg@sc.edu)  
[baoj@cec.sc.edu](mailto:baoj@cec.sc.edu)

Submitted to:

Jeniffer Becker  
ARMY RESEARCH OFFICE  
P.O. BOX 12211  
RESEARCH TRIANGLE PARK, NC 27709-2211

Date September 23, 2013

Project Number: W911NF-11-1-0210

Capability Area: CB filtration

### **Executive summary**

The impregnated active carbon used in air purification systems degrades over time due to exposure to contamination and mechanical effects (packing, settling, flow channeling, etc.). To estimate the residual life of the carbon filter, many methods have been developed to detect the changes of various parameters of the carbon bed [1][2]. A previous study has shown that the electrochemical impedance spectroscopy (ECIS) of carbon bed can be used to detect water vapor condensation and acid forming gas in carbon bed [3]. However, it's difficult to differentiate the impedance changes due to chemical contamination from those due to mechanical changes. In this project a novel approach is proposed to use piezoelectric wafer active sensors (PWAS) embedded into the carbon bed to evaluate the overall chemical and mechanical conditions of the carbon bed during SO<sub>2</sub> contamination tests. On one hand, the electromechanical impedance spectroscopy (EMIS) of the PWAS indicates the mechanical impedance changes of the carbon bed; on the other hand, the ECIS of the PWAS indicates the conductivity change of the carbon bed due to the formation of sulfuric acid during the SO<sub>2</sub> contamination.

This is a novel detection approach that combines two different methods and requires the study of fundamental science issues. Some remarkable new phenomena are unveiled during the project:

1. PWAS EMIS change during the carbon bed SO<sub>2</sub> contamination test
2. PWAS EMIS can monitor changes in mechanical pressure
3. PWAS EMIS can detect temperature changes inside the carbon bed
4. PWAS EMIS can detect the presence of contaminants, such as water and kerosene in the carbon bed
5. EMIS and ECIS measurements are consistent with each other and complimentary

A simplified impedance model was created to simulate the PWAS-carbon bed system under increasing pressure. Similar impedance change pattern was observed when comparing the simulation results with experimental data. A series of hardware and software are developed to carry out the experiments and data analysis. A carbon bed contamination apparatus is designed and constructed to perform the SO<sub>2</sub> contamination experiments. A carbon bed tester is developed to host the carbon granules, the PWAS transducers, and the temperature sensors. The carbon tester is also able to change the pressure applied to the carbon bed during the experiments. A data analysis method was developed to extract the small variation of the PWAS EMIS peak. MATLAB and LabVIEW software were developed to assist the carbon bed SO<sub>2</sub> contamination test, the PWAS EMIS signal acquisition, and the data analysis.

At the end of the project, we have achieved our goal as presented in the project proposal:

1. Develop a PWAS-carbon bed impedance model, mechanical and chemical degradations that are relevant to carbon filter residual life estimation will be considered
2. Use specialized experiments to validate and verify the model
3. Develop data analysis methods and residual life estimation methodology
4. Design and construct a bench top demonstrator to test the overall methodology
5. Specify the requirement for a proof of concept prototype development and write up the final report

Through the project, we have established the fundamental knowledge about how the PWAS transducers EMIS respond to the chemical and mechanical changes in the carbon bed. To move forward towards a practical carbon bed residual life estimator, future research is suggested to focus on the predictive modeling of the interaction between the PWAS and the carbon bed. Both analytical and finite element method (FEM) can be utilized to quantitatively predict the behavior of PWAS EMIS under various carbon bed chemical and mechanical conditions. Specialized experiments should be designed to isolate the irrelevant parameters, such as the pressure level, the temperature, and the conductivity of the carbon granules.

During the project, we have reported regularly to the program manager and the technical specialists of the sponsor agency. Through their consultation and guidance, we have gained much insight and knowledge in various aspects of the carbon bed contamination behavior. Their support is gratefully acknowledged.



## Statement of the problem studied

The impregnated active carbon used in air purification systems degrades over time due to exposure to contamination and mechanical effects (packing, settling, flow channeling, etc.). To estimate the residual life of the carbon filter, many methods have been developed to detect the changes of various parameters of the carbon bed [1][2]. A previous study has shown that the electrochemical impedance spectroscopy (ECIS) of carbon bed can be used to detect water vapor condensation and acid forming gas in carbon bed [3]. However, it's difficult to differentiate the impedance changes due to chemical contamination from those due to mechanical changes. In this project a novel approach is proposed to use piezoelectric wafer active sensors (PWAS) embedded into the carbon bed to evaluate the overall chemical and mechanical conditions of the carbon bed during SO<sub>2</sub> contamination tests. On one hand, the electromechanical impedance spectroscopy (EMIS) of the PWAS indicates the mechanical impedance changes of the carbon bed; on the other hand, the ECIS of the PWAS indicates the conductivity change of the carbon bed due to the formation of sulfuric acid during the SO<sub>2</sub> contamination.

## Summary of the most important results

### 1. Summary for the October 2011 – July 2012 reporting period

In this reporting period, our research has three main branches: a) on the theoretical modeling of PWAS interaction with carbon bed; b) experimental study on PWAS impedance change during SO<sub>2</sub> contamination; and c) data analysis, presentation, and management procedures development and software programming.

In the **theoretical modeling** branch, we have improved our previous simplified coupled PWAS-carbon bed impedance model by extending to 2-D circular PWAS electromechanical model, and improve the PWAS-carbon bed interaction model by adding damped boundary conditions. We studied the carbon granule resistance pressure effects through literature survey and experiments; and the temperature effects on impedance through literature survey and experiments.

In the **experimental study** branch, we have improved our testing system and syringe tester to adapt to the SO<sub>2</sub> tests. We have improved the design of our syringe tester to incorporate multiple sensors in each test. We conducted substantial number of experiments at different carbon bed pressure, SO<sub>2</sub> contamination level, with insulated and non-insulated PWAS.

In the **data analysis and software development** branch, we have developed data metrics to present the effects of applied pressure level, and SO<sub>2</sub> contamination level effects on the PWAS EMIS. We also developed software to help us managing, categorizing and comparing data with different mechanical and chemical factors.

Through data analysis, we found different response patterns from insulated PWAS and non-insulated PWAS. The data analysis results verified our concepts of: 1) using PWAS impedance to **detect pressure and SO<sub>2</sub> loadings** on carbon bed; 2) insulated PWAS and non-insulated PWAS **can differentiate pressure and SO<sub>2</sub> effects**.

A **patent application** was filed to the USPTO with the title "Methods and Sensors for the Detection of Active Carbon Filters Degradation with EMIS-ECIS PWAS" on December 1, 2011(serial# 13/309,149).

### **Key achievements**

1. Improved our theoretical PWAS-carbon bed impedance model.
2. Designed and constructed combined pressure-SO<sub>2</sub> testing device for PWAS-carbon bed impedance.
3. Experimental data analysis verifies our concepts on PWAS EMIS-ECIS detecting and differentiating mechanical and chemical loadings on carbon bed.
4. Patent application was filed to the USPTO with the title "Methods and Sensors for the Detection of Active Carbon Filters Degradation with EMIS-ECIS PWAS" (serial# 13/309,149).

### 2. Summary for the August 2012 - July 2013 reporting period

In this reporting period, our main focus was on **developing the experimental method** for detecting SO<sub>2</sub> contamination level in the carbon bed with embedded piezoelectric wafer active sensors (PWAS) using electromechanical impedance spectrum (EMIS) and electrochemical impedance spectrum (ECIS). By applying the theoretical and experimental results obtained from previous reporting period, we designed a series of carbon bed SO<sub>2</sub> contamination experiments. During the experiments, high concentration SO<sub>2</sub> was diluted with air, and the

mixture flow through carbon bed embedded with PWAS transducers. The SO<sub>2</sub> breakthrough was detected with titration method by running the exhaust pipe from the carbon bed through an H<sub>2</sub>O<sub>2</sub> bath. The impedance spectrum of the PWAS transducers inside the carbon bed was collected and analyzed. We were able to achieve various mechanical pressure loading levels, humidity level, and SO<sub>2</sub> contamination levels in the carbon bed. The PWAS resonance EMIS peak frequency and amplitude shows distinctive change patterns in response to the mechanical and chemical loading conditions.

The main research developed multiple branches during the course of development. The major branches include **PWAS transducers characterization, PWAS fabrication techniques**, and design a **brass board demonstrator** for our detection method. The findings from these studies helped us to improve the experimental design and adjust the testing procedures.

Efforts were made to improve the experimental environment and testing capabilities. We negotiated with the chemical engineering department in University of South Carolina, and **secured a chemical lab for future SO<sub>2</sub> contamination experiments**. We established discussion channels with multiple PWAS suppliers to **improve the PWAS design** and to achieve **higher production quality standard**. We collaborated with physics department in University of South Carolina to experiment several novel **PWAS fabrication techniques**. We also worked with multiple impedance analyzer suppliers to evaluate the performance of several impedance analyzers. A **portable impedance analyzer** was purchased based on the evaluation results.

In the beginning of this year, we also **visited the ECBC labs** to discuss our research findings and to learn the ECBC carbon bed contamination experimental setup and testing procedures. With the input from ECBC, we have improved our experimental techniques, testing parameters, and brass board design.

#### ***Key achievements***

1. developed an experimental method for detecting SO<sub>2</sub> contamination level in the carbon bed with embedded piezoelectric wafer active sensors using electromechanical and electrochemical impedance spectrum
2. achieved various mechanical pressure loading levels, humidity levels, and SO<sub>2</sub> contamination levels in the carbon bed
3. the PWAS resonance EMIS peak frequency and amplitude shows distinctive change patterns in response to the mechanical and chemical loading conditions
4. designed a brass board to demonstrate the detection method

#### **3. Summary for the August 2013 - July 2014 reporting period**

In this reporting period, we focused on two major activities: (a) improving the PWAS EMIS tester design, the test procedure, and (b) experimental validation of the PWAS EMIS tester setup and the data analysis method. At the end of this reporting period, we have finalized the EMIS tester setup design and construction. We have performed multiple experiments to validate the PWAS EMIS testing procedure. The data analysis method was examined with the experimental data.

We have integrated the mass flow controllers into the carbon bed contamination setup. The mass flow controllers are able to control the flow of N<sub>2</sub> and SO<sub>2</sub> into the carbon bed. We have performed several experiments to adjust the testing setup. An important modification to the system is to include a purge line from the N<sub>2</sub> cylinder to the SO<sub>2</sub> route to flush the SO<sub>2</sub> mass flow controller after experiments. By reviewing the data from multiple experiments, improvement of the experimental procedures are made: such as reducing the frequency range and increasing the frequency resolution when collecting the impedance data. We have adjusted the frequency range to cover only the first PWAS resonance peak (200 kHz-400 kHz), and increase the frequency resolution (0.1 kHz) to provide better peak quality for the analysis.

The mechanical characters of the carbon bed change slowly during the experiment. Hence, the PWAS EMIS spectrums often have only very small variations between samples. A data analysis method is experimented to capture the trend of change in the EMIS spectrum. This method is similar to the full width at half maximum (FWHM) method implemented in the fiber Bragg grating (FBG), where the intensity of the light reflected by the FBG at the half peak frequency is used to detect the strain change in the FBG.

### Bibliography

- [1] Bernard, P., Caron, S., St.Pierre, M., and Lara, J., “*End-of-service indicator including porous waveguide for respirator cartridge*,” Institut National D'Optique, Quebec, Patent No. US6375725 (2002)
- [2] Hori, H., Ishidao, T., and Ishimatsu, S., “*Development of a new respirator for organic vapors with a breakthrough detector using a semiconductor gas sensor*,”. Applied Occupational and Environmental Hygiene, 18, 90-95 (2003).
- [3] Rubel, G. O., Peterson, G. W., Fletcher, N. K., Parker, J. E. & Jeffers, R. B. “Measurement of the impedance change of impregnated activated carbon during exposure to SO<sub>2</sub> vapors at ambient temperatures”, Carbon, 47, 3566-3573, (2009)
- [4] Giurgiutiu, V., *Structural Health Monitoring with Piezoelectric Wafer Active Sensors*, Elsevier Academic Press, 760 pages, ISBN 978-0120887606, (2008)
- [5] Bao, J.; Giurgiutiu, V.; Rubel, G.; Peterson, G.; Ball, T. “Active Carbon Filter Health Condition Detection with Piezoelectric Wafer Active Sensors”, *Proceedings of the 2011 SPIE Smart Structures/NDE*, March 7-11, 2011, San Diego, CA
- [6] Xu, B. and Giurgiutiu, V. “*Development of DSP-based Electromechanical (E/M) Impedance Analyzer for Active Structural Health Monitoring*,” In SPIE's 13th International Symposium on Smart Structures and Materials and 11th International Symposium on NDE for Health Monitoring and Diagnostics, Health Monitoring and Smart NDE of Structural and Biological Systems Conference, eds. M. Tomizuka, C.-B. Yun & V. Giurgiutiu. San Diego, CA USA, (2006)
- [7] Giurgiutiu, V., Friedman, H., Bender, J., Borg, T., Yost, M. J., Newcomb, W., et al., “*Electromechanical impedance sensor for in vivo monitoring the body reaction to implants*,” Journal of Investigative Surgery, 17, 257-270. doi: 10.1080/08941930490502835 (2004)
- [8] Giurgiutiu, V., “*In-Situ Structural Health Monitoring, Diagnostics and Prognostics System Utilizing Thin Piezoelectric Sensors*” Patent No. US 7, 024, 315 B2, Date of Patent Apr. 42006 (2006)
- [9] Giurgiutiu, V., Bao, J., Rubel, G., Peterson, G., “*Method and Sensor for the Detection of Active Carbon Filters Degradation with EMIS-ECIS PWAS*” USC-IPMO, Disclosure ID No. 00872 (2010)

# **Quarterly REPORT**

**For the period October - December 2013**

## **PWAS EMIS-ECIS Active Carbon Filter Residual Life Estimation Methodology**

Submitted by:

Victor Giurgiutiu and Jingjing Bao  
University of South Carolina  
300 Main Street, Columbia SC 29208  
Telephone: (803) 777-8018  
Email: victorg@sc.edu

Submitted to:

Gregory W. Peterson  
CBR Filtration Branch  
U. S. Army Edgewood Chemical Biological Center  
5183 Blackhawk Road, Bldg. 3549  
APG, MD 21010  
Telephone: (410) 436-9794  
Email: gregory.w.peterson@us.army.mil

Date Dec 23, 2013

Project Number: W911NF\_11\_1  
Capability Area: CB filtration

## Contents

1	Project and report overview .....	2
2	Progress during the reporting period (October-December 2013).....	3
2.1	Evaluate new PWAS transducer sample performance .....	3
2.1.1	Statistical study on the PWAS samples of improved design .....	3
2.1.2	Literature survey on PWAS transducer characterization and impedance feature extraction .....	7
2.1.3	Suggestions on the incoming PWAS sorting procedure .....	9
2.2	Build the new carbon bed SO <sub>2</sub> contamination testing apparatus .....	9
2.2.1	Improving the design of the carbon bed SO <sub>2</sub> contamination testing apparatus.....	9
2.2.2	Suggestion on Ion chromatography testing procedure.....	11
3	Planned activities for next quarter (January-March 2014).....	11
4	References .....	12

### 1 PROJECT AND REPORT OVERVIEW

In this reporting period, we focused on two tasks: (a) evaluate new PWAS transducer sample performance; and (b) build the new carbon bed SO<sub>2</sub> contamination testing apparatus.

#### **Evaluate new PWAS transducer sample performance**

In the previously reported PWAS transducer characterization study, we measured the mechanical, electrical, and electromechanical impedance spectroscopy (EMIS) parameters of multiple PWAS samples made by our current PWAS supplier; a statistical study was carried out to find the distribution of these sensor parameters. A wide spread was found in the measured data. The conclusion was that **we need to improve the PWAS consistency** to make it possible to detect minimal changes during the SO<sub>2</sub> contamination of carbon bed.

In this reporting period, we have purchased 25 samples from another PWAS supplier. The PWAS transducer design was adjusted to have (a) circular shape, and (b) center soldered wires to improve the center symmetry, hence reduce the complexity of the impedance spectrum. The dimension, capacitance, and impedance spectrum were measured to conduct a **statistical study on the new PWAS transducer performance parameters**. Mix results were obtained from the study: while better impedance peak shape were observed, the consistency of the impedance peak frequency and amplitude was not desirable.

After the two rounds of statistical study of the PWAS transducer from two different PWAS suppliers, we found it's necessary to establish an incoming PWAS **sorting procedure** to improve the PWAS transducers consistency. The procedure should be able to separate sensors into several "bins" base on their impedance characteristics. Literature survey was conducted on PWAS transducer characterization and impedance feature extraction. Several parameter indicators are suggested in this report to establish the sorting procedure.

#### **Build the new carbon bed SO<sub>2</sub> contamination testing apparatus**

In the previous reporting period, we reported the improved design of the carbon bed SO<sub>2</sub> contamination testing apparatus. The improvement include: using a lower SO<sub>2</sub> concentration (0.5% SO<sub>2</sub>+99.5%N<sub>2</sub>) level, introducing adjustable humidity level to the N<sub>2</sub> diluent, monitoring carbon bed temperature, etc. To **build the improved design**, changes to current components are needed, such as: low concentration SO<sub>2</sub> cylinders, N<sub>2</sub> cylinders, accessories for the new cylinders. New components are also needed, such as a saturator to introduce humidity to the diluent. We also introduced a pH meter to assist the titration procedure. In this reporting period, new components started arriving from the suppliers, and we started building the new carbon bed SO<sub>2</sub> contamination testing apparatus.

We consulted with a couple of chemical testing labs on precision measurement of different chemical detection methods that can be utilized for the qualification of SO<sub>2</sub> absorbed in the carbon bed. Through discussion, we found that ion chromatography can be utilized to assist detection and quantification of SO<sub>2</sub> in the carbon bed. **Ion**

**chromatography testing procedure** is suggested in the report. We have targeted two labs for the upcoming experiment, and will experiment with the procedure once approved by the ECBC specialists.

The progress details are covered in the following sections.

## 2 PROGRESS DURING THE REPORTING PERIOD (OCTOBER-DECEMBER 2013)

### 2.1 Evaluate new PWAS transducer sample performance

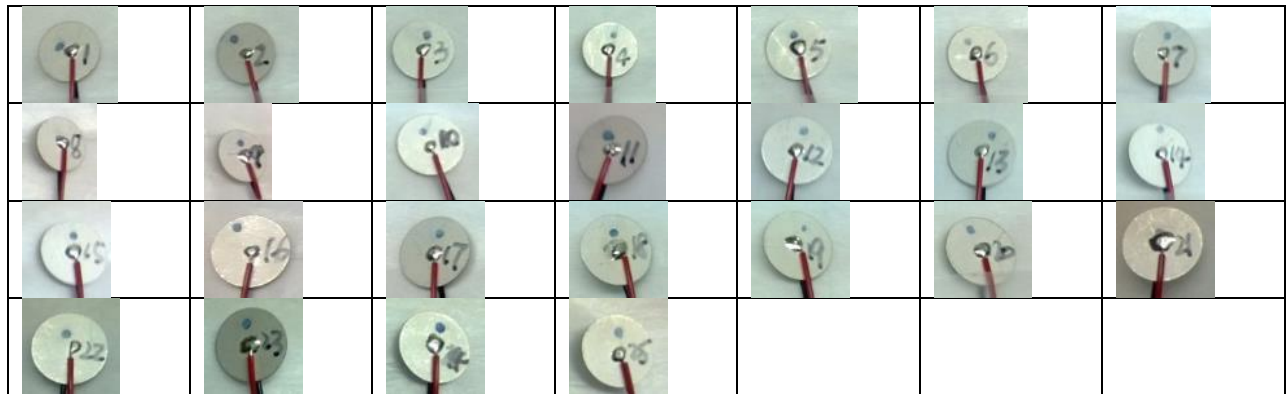
In the previously reported PWAS transducer characterization study, we measured the mechanical, electrical, and electromechanical impedance spectroscopy (EMIS) parameters of multiple PWAS samples made by our current PWAS supplier; a statistical study was carried out to find the distribution of these sensor parameters. A wide spread was found in the measured data. The conclusion was that **we need to improve the PWAS consistency** to make it possible to detect minimal changes during the SO<sub>2</sub> contamination of carbon bed.

In this reporting period, we have purchased 25 samples from another PWAS supplier. The PWAS transducer design was adjusted to have (a) circular shape, and (b) center soldered wires to improve the center symmetry, hence reduce the complexity of the impedance spectrum. The dimension, capacitance, and impedance spectrum were measured to conduct a **statistical study on the new PWAS transducer performance parameters**. Mix results were obtained from the study: while better impedance peak shape were observed, the consistency of the impedance peak frequency and amplitude was not desirable.

#### 2.1.1 Statistical study on the PWAS samples of improved design

In this reporting period, we received the new PWAS samples (from APC international ltd.) with pre-soldered wires. Before placing the order, we discussed with the supplier regarding how to improve the PWAS transducer impedance spectrum consistency. Two main factors were taken into account in the final design of the new samples:

- 1) use **circular** PWAS to create a single first resonance peak, which is defined by the diameter of the circle
- 2) solder the electrical wire to the **center** of the transducer to improve the mass distribution symmetry



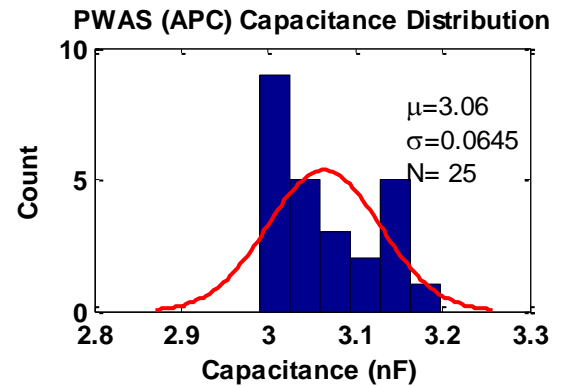
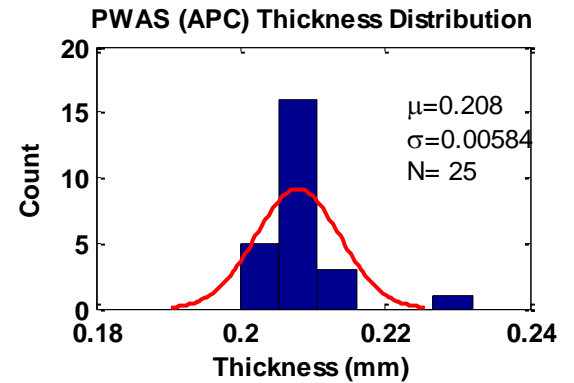
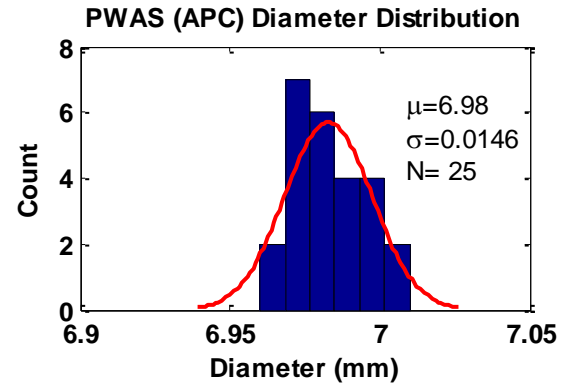
**Figure 1 New PWAS samples with pre-soldered wires.**

When the samples arrived, we first conducted a visual inspection on the sensor quality and soldering point quality. Figure 1 shows pictures of the new samples. During visual inspection, we found the qualities of the soldering points on the new PWAS samples are not as desirable. The variation of the size and shape of the soldering point are obvious across the 25 samples.

The first step of quantitatively characterization of the PWAS transducers is to measure the dimensions and capacitance of the transducers. Table 1 shows the data measured from the 25 samples. The data distributions of the diameter, thickness and capacitance values are also presented with the data table. From the distribution charts, it seems that the diameter distribution is reasonably close to normal distribution; while there are a few outliers on the thickness distribution; and the capacitance is positively skewed (tail at the right).

**Table 1 PWAS sample dimension and capacitance**

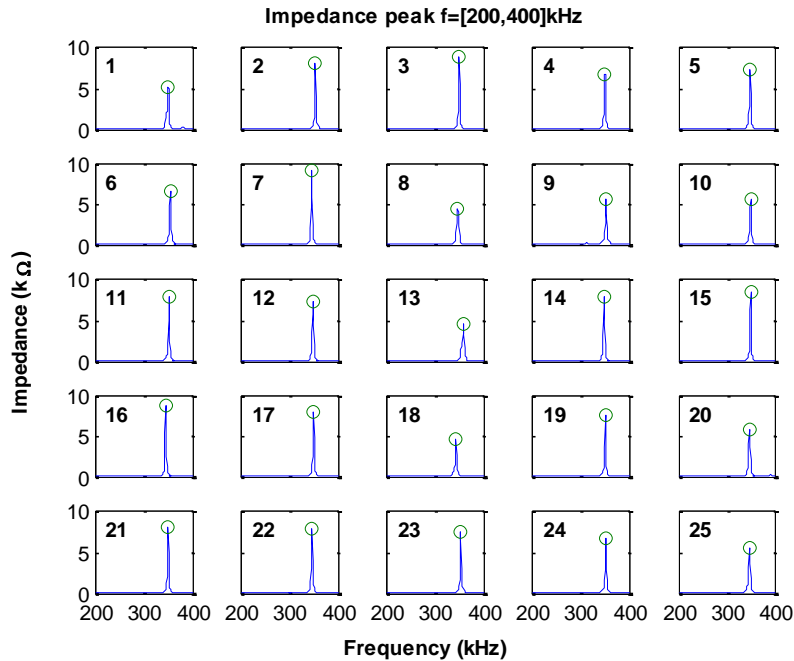
PWAS	Diameter (mm)	Thickness (mm)	Capacitance (nF)
1	6.97	0.204	3.104
2	6.97	0.206	3.027
3	6.97	0.208	3.112
4	6.97	0.207	2.998
5	6.98	0.208	3.047
6	6.96	0.211	3.022
7	7.00	0.207	3.062
8	6.98	0.206	3.021
9	6.96	0.203	3.001
10	6.99	0.208	2.991
11	6.98	0.207	3.046
12	6.99	0.205	3.078
13	6.97	0.207	2.995
14	6.97	0.206	3.028
15	7.00	0.209	3.146
16	7.01	0.210	3.015
17	7.01	0.200	3.158
18	6.98	0.212	3.198
19	6.99	0.232	3.162
20	6.97	0.206	2.990
21	6.98	0.208	3.038
22	7.00	0.208	3.065
23	6.99	0.201	3.156
24	7.00	0.206	3.139
25	6.98	0.213	2.996



After these basic measurements, we tested the admittance and impedance of the samples with the HP-4194A impedance analyzer, and plotted the resonance peaks in two frequency bands, as shown in Figure 2, and Figure 3.

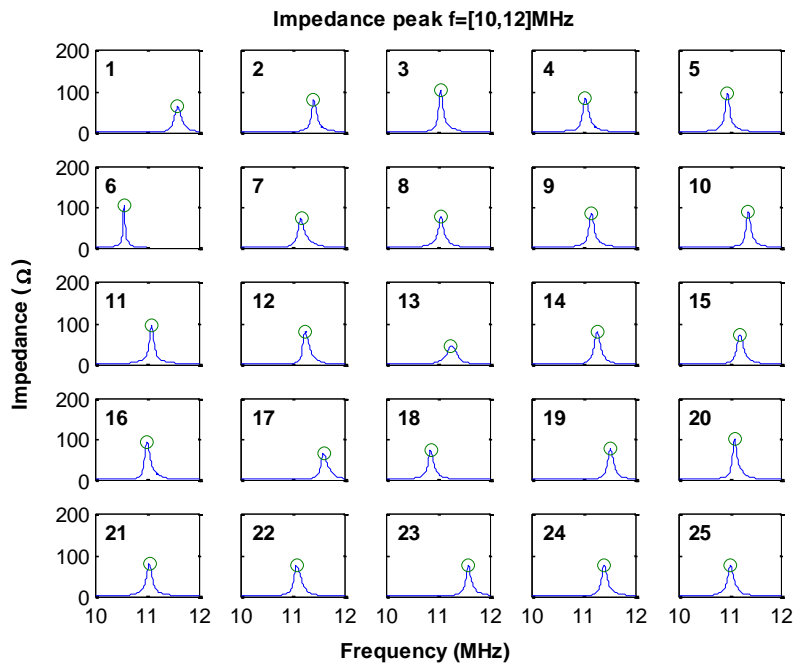
It is apparent that at the low frequency band (200-400 kHz) range, there is only one impedance peak. This peak is due to the dominant vibration mode that is symmetric to the center of the circle. Because the transducer is of circular shape, and the wires were soldered to the center of the PWAS, the transducer vibrates in a mode close to symmetric to the center of the circular shape. For the previously reported rectangular shaped PWAS, in comparison, both the length and width produced low frequency impedance peaks; and the wires soldered to the corner of the transducer, introduced unbalanced mass, which also affected the impedance peak shape and frequency.

For the higher frequency (10-12 MHz) range, the impedance peak was also nice and clean. This peak is related to the PWAS vibration mode in the thickness direction. It is apparent that the peak frequency and amplitude are not very consistent.



**Figure 2 Impedance peak of PWAS samples around the first resonance mode frequency.**

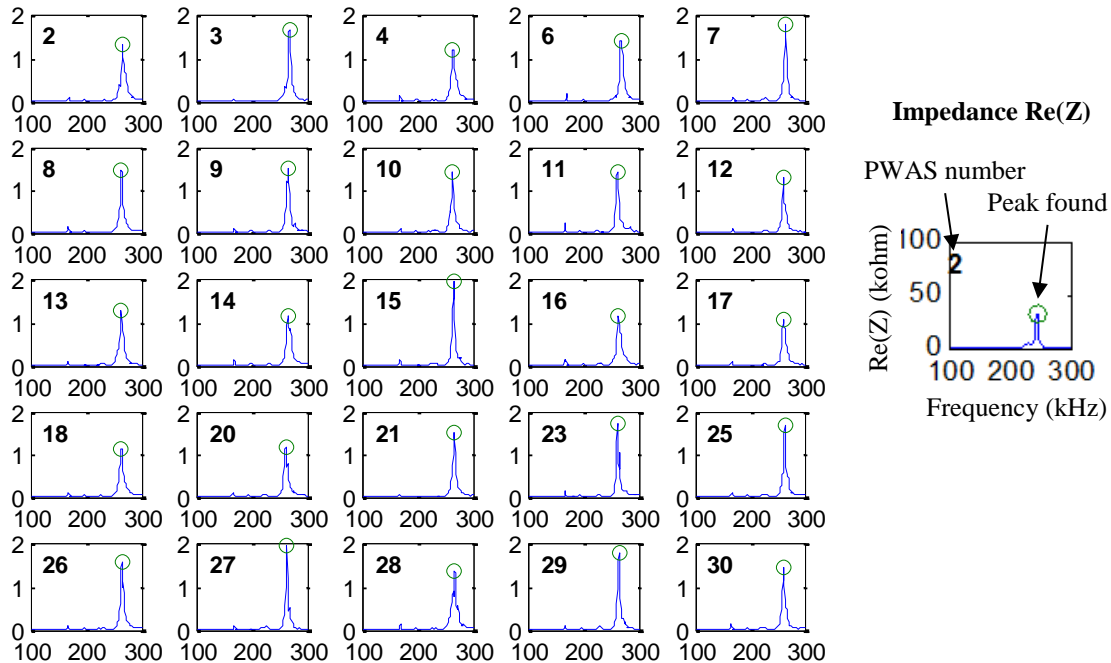
From the



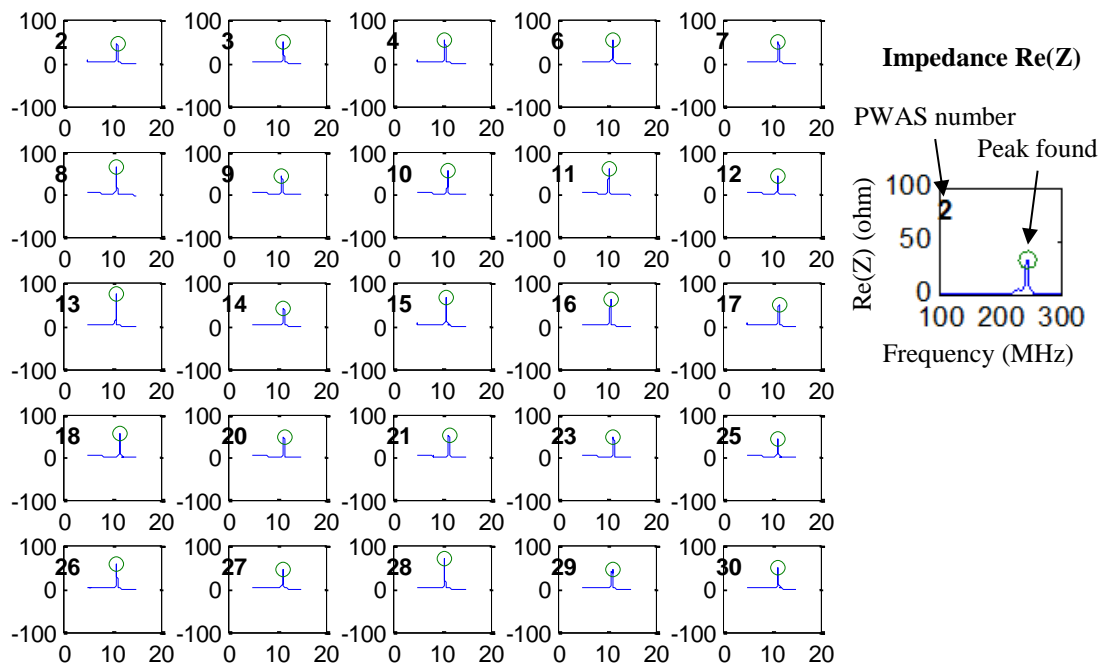
**Figure 3 Impedance peak of PWAS samples around the thickness resonance mode frequency.**

We compare these data from the new PWAS samples with the previously reported data from the rectangular shaped PWAS transducers. The previously reported impedance spectrum of the rectangular shaped transducers is presented in Figure 4 and Figure 5. In Figure 4, there are several small amplitude peaks appear at frequencies lower than the large resonance peak at around 260 kHz. It is also obvious that the peak amplitude of the circular PWAS is much larger than those obtained from the rectangular PWAS transducers.





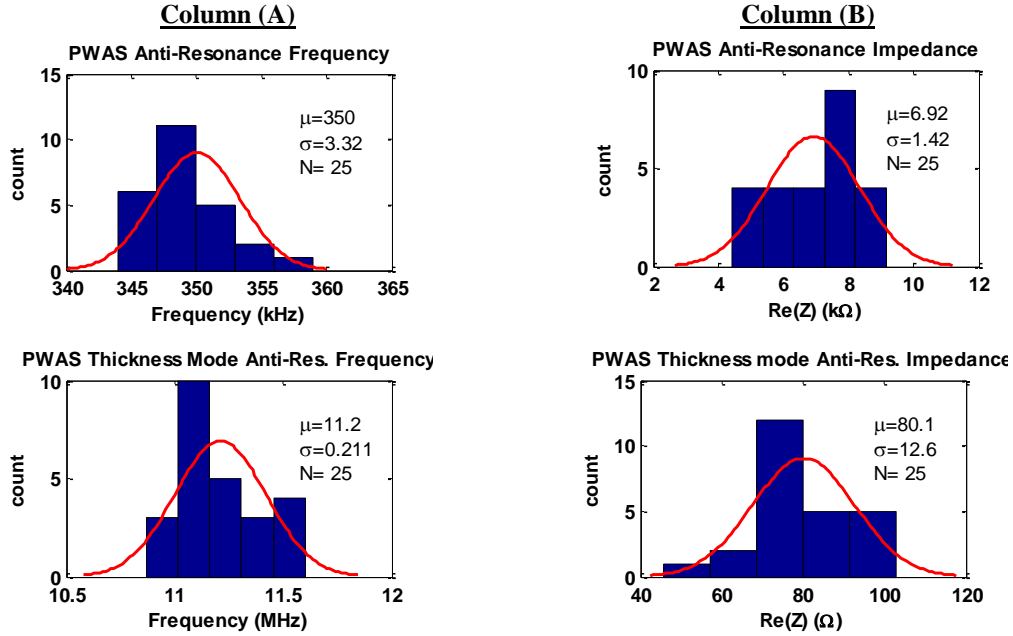
**Figure 4 Test 2: Impedance spectrum frequency and amplitude of the first in-plane resonance peak**



**Figure 5 Test 2: Impedance spectrum frequency and amplitude of the thickness resonance peak**

In summary, we have the following observations:

- 1) With the new PWAS sample, an apparent improvement is that the first resonance mode has single peak, while the previously reported PWAS samples has multiple peaks.
- 2) On the other hand, the discrepancy of the peak frequency, amplitude, and shape are can be noticed. The variation of the soldering quality, as shown in Figure 4, may be the reason for such discrepancy.



**Figure 6** Statistical analysis of the PWAS EMIS data: column (A) frequency distribution parameters, and column (B) peak value distribution parameters

Furthermore, we performed a statistical analysis with the EMIS data sets from the 25 samples. Figure 6 shows the histogram of the impedance peaks frequency and amplitude distributions. To evaluate whether or not the new samples present improved consistency, we compared the statistical parameters between the previously reported rectangular shaped samples and the new circular shaped samples, as shown in Table 2

**Table 2** Comparison of the resonance peak distribution parameters between previously reported rectangular PWAS samples and new circular PWAS samples

	First resonance mode				Thickness resonance mode			
	Frequency(kHz)		Amplitude (kΩ)		Frequency(MHz)		Amplitude(Ω)	
	$\mu$	$\sigma$	$\mu$	$\sigma$	$\mu$	$\sigma$	$\mu$	$\sigma$
Rectangular samples (previous)	264	2.24	1.49	0.241	11.2	0.32	52.2	9.34
Circular samples (new)	350	3.32	6.92	1.42	11.2	0.211	80.1	12.6

It can be noticed that the amplitude of the peaks are larger for the new samples while there is no clear improvement on the parameter variations. In fact, the parameters spread are more than that of the previous samples.

### 2.1.2 Literature survey on PWAS transducer characterization and impedance feature extraction

The usage of PWAS EMIS signal in this research is analogue to their usage in structural health monitoring (SHM) applications. The characterization and impedance feature extraction methods are essential to sense the minimal changes introduced by the carbon bed contamination. To this purpose, good signal processing and signal denoising algorithms are essential for successful detection. The PWAS EMIS approach resembles modal analysis which relies on vibration spectra and modal parameters (resonance frequencies, peak amplitudes, modal damping, etc.). Both modal testing and E/M impedance spectroscopy (EMIS) produce frequency spectra that need to be analyzed in order

to detect changes in the structural state. The physical support of these methods is the fact that structural change, such as fatigue cracks in metals or delamination of layer composite structures in SHM applications, causes characteristic local changes of stiffness, damping, and/or mass [1][2]. In this research, the carbon bed condition change may cause similar effects. Corresponding to these changes, shifts of the dynamic characteristics (frequencies, mode shapes, modal damping) occur. The deviation between the pristine dynamic properties and the damaged dynamic properties can be used to detect damage, and diagnose its location and extent.

In the SHM applications, the word "damage" represents the changes in the structure underneath the PWAS transducers. In this application, similarly, we can use the word "damage" to represent the change in the carbon bed surrounding the PWAS transducers. The damage identification algorithms should separate PWAS EMIS data into several classes depending on the damage severity and/or location (e.g., pristine, lightly damaged, severely damaged, etc.). When a human analyst compares the frequency spectra of pristine and damaged structures, the difference between spectra may be obvious in many cases, i.e., "it can be seen with the naked eye". Doing the same process automatically requires a classification algorithm which may not be as easy to build.

Damage identification algorithms are vital for practical implementation of SHM system. For successful damage identification during an SHM process, the deviation of inherent parameters of the structure due to temperature, humidity, or integrity changes as reflected in the measured data should be accounted for [3]. The simplest damage identification algorithm is the 'damage metric' a.k.a. 'damage index', 'damage indicator', 'DI', which is a formula that is applied to the whole signal and yields a scalar value, i.e., a mere number. This scalar value serves as a measure of the amount of damage present in the structure. In spectrum based SHM, the damage metric results from the comparative processing of 'pristine' and 'damage' spectra. The damage metric should be able to identify the difference between the two spectra due to damage presence. A good damage metric should only capture the spectral features that are directly modified by the damage presence, whereas neglecting the variations due to normal operation conditions (i.e., statistical difference within a population of specimens, and expected changes in temperature, pressure, ambient vibrations, etc.). Ideally, the damage metric should be able to evaluate the vibration or E/M impedance spectra and indicate damage presence, location, and severity. Development of suitable damage metrics and damage identification algorithms remains an open question in the practical application of structural health monitoring methods.

To date, several damage metrics have been proposed and tried; among them, the most popular are the root mean square deviation (RMSD), the mean absolute percentage deviation (MAPD), and the correlation coefficient deviation (CCD). The mathematical expressions for these metrics are as follows

$$RMSD = \sqrt{\frac{\sum (S_i - S_i^0)^2}{N}}{\sqrt{\sum (S_i^0)^2}}, \quad (1)$$

$$MAPD = \sum \left| \frac{S_i - S_i^0}{S_i^0} \right|, \quad (2)$$

$$Cov = \frac{1}{N-1} \sum (S_i - \bar{S})(S_i^0 - \bar{S}^0) \quad (3)$$

$$CCD = 1 - \frac{\sum (S_i - \bar{S})(S_i^0 - \bar{S}^0)}{\sqrt{\sum (S_i - \bar{S})^2 \sum (S_i^0 - \bar{S}^0)^2}}, \quad (4)$$

where  $N$  is the number of frequencies in the spectrum,  $S_i$  are the elements of the spectrum, and the superscript 0 signifies the pristine state of the structure. The symbols  $\bar{S}$ ,  $\bar{S}^0$  signify mean values.

Equations (1)-(4) yield a scalar number, the damage index (DI), which represents the relationship between the compared spectra. Thus, we expect that the resonant frequency shifts, the peaks splitting, and the appearance of new resonances that appear in the spectrum will alter the DI value and thus indicate the presence of damage. The advantage of using Eqs. (1)-(4) is that the spectrum does not need any pre-processing, i.e., the data obtained from the measurement equipment can be directly used to calculate the DI.

In this research, because the minimal change in the carbon bed during the contamination process, the shift of peak frequency and change of peak amplitude is expected to be very small. It is desirable to have PWAS transducers with similar impedance peak parameters to be used in a set of experiments, such that small changes can be identified. If the initial impedance peaks have much difference from transducer to transducer, the definition of an algorithm base on the impedance frequency and amplitude would be very difficult. By using the DI defined in equations (1)-(4), it is possible to define an algorithm, which will find the similarity among multiple PWAS transducers, and provide a scalar number to present the degree of similarity.

### 2.1.3 Suggestions on the incoming PWAS sorting procedure

After the two rounds of statistical study of the PWAS transducer from two different PWAS suppliers, we found it's necessary to establish an incoming sensor **sorting procedure** to improve the consistency of PWAS transducers to be used in a group of experiments. The procedure should be able to separate sensors into several "bins" base on their impedance characteristics.

Base on the literature survey in the previous section, a possible sorting procedure is list as following:

1. When the PWAS transducers are received, the dimension and capacitances of each transducer can be measured.
2. The impedance spectrum of each transducer can be captured using impedance analyzer and recorded for later reference.
3. By browsing the impedance peaks recorded in step 2, a "standard" sensor can be selected to present the desirable sample. This sample will be used as the "baseline" for selecting the sensors.
4. Comparing each sample's impedance spectrum with the "baseline" data selected in step 3 using the "damage index" (equations (1)-(4) defines a few candidates), the similarity of each sample to the "standard" sample can be evaluated.
5. A threshold of "damage index" can be applied find the samples that are most close to the standard sample.
6. Repeat steps 3-5 for the rest of the samples.

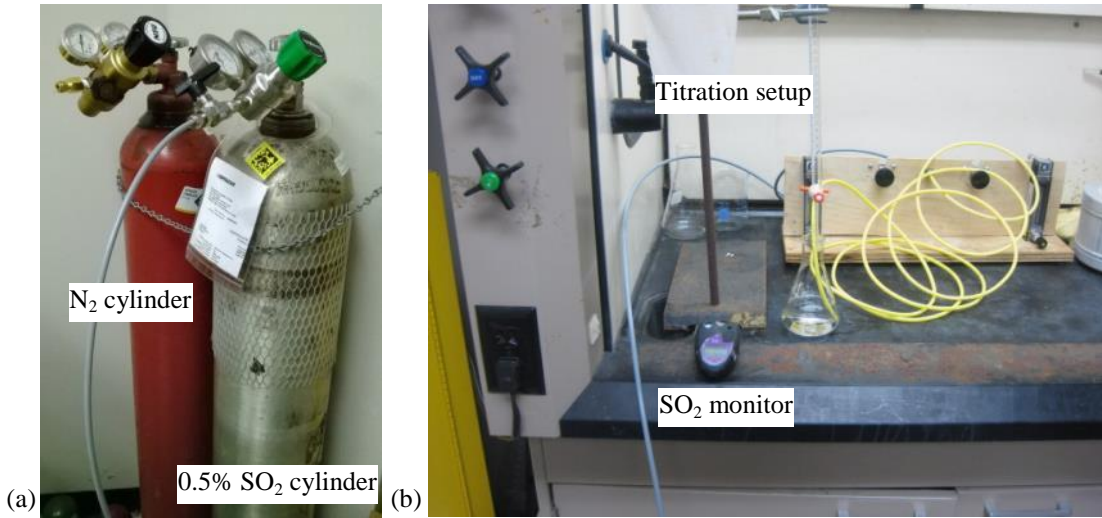
It is important to note that the damage index algorithm has been successfully applied in SHM applications to identify the changes of a PWAS with its **own** baseline, not to compare **different** PWAS transducers. This is only a preliminary idea of the sorting procedure. Extensive experiments and data analysis need to be carried out to validate the concept. In the next reporting period, we plan to carry out more study on the characterization of PWAS transducers, and will evaluate the performance of damage index concept in sorting the PWAS transducers.

## 2.2 Build the new carbon bed SO<sub>2</sub> contamination testing apparatus

In the previous reporting period, we reported the improved design of the carbon bed SO<sub>2</sub> contamination testing apparatus. The improvement include: using a lower SO<sub>2</sub> concentration (0.5% SO<sub>2</sub>+99.5%N<sub>2</sub>) level, introducing adjustable humidity level to the N<sub>2</sub> diluent, monitoring carbon bed temperature, etc. To **build the improved design**, changes to current components are needed, such as: low concentration SO<sub>2</sub> cylinders, N<sub>2</sub> cylinders, accessories for the new cylinders. New components are also needed, such as a saturator to introduce humidity to the diluent. We also introduced a pH meter to assist the titration procedure. In this reporting period, new components started arriving from the suppliers, and we started building the new carbon bed SO<sub>2</sub> contamination testing apparatus.

### 2.2.1 Improving the design of the carbon bed SO<sub>2</sub> contamination testing apparatus

Figure 7 shows the experimental setup in the lab. SO<sub>2</sub> cylinder and N<sub>2</sub> cylinder were properly installed in the lab with an additional valve connected to the output port of the regulator on the 0.5% SO<sub>2</sub> tank as an emergency shut precaution. A SO<sub>2</sub> detector (Honeywell Analytics 54-45-03VD SO<sub>2</sub> 0-100 ppm measuring range) will be used for monitoring the SO<sub>2</sub> level outside the fume hood to protect operators from exposure to SO<sub>2</sub>.



**Figure 7** Experimental setup for the low concentration SO<sub>2</sub> experiments include (a) the 0.5% SO<sub>2</sub> cylinder and N<sub>2</sub> cylinder; and (b) the flow control and titration setup inside the fume hood, and SO<sub>2</sub> detector for monitoring the SO<sub>2</sub> level to protect operator from exposure to SO<sub>2</sub>.

To introduce a controllable humidity level into the diluent, we have been in contact with the saturator manufacturer to finalize the saturator's design. By calculation, we found that the maximum amount of water required for a 90-minute test with 85% RH level is a little over 5 liters. The finalized design is shown in Figure 8.

Scenario D	15%RH	80%RH
V/V in bottle	0.5	0.5
total flow rate (l/min)	2.197	2.197
chemical flow rate ml/min	267	267
carbon mass (g)	8	8
test time (min)	90	90
Volume Fraction H <sub>2</sub> O in Nitrogen	0.0313	0.0313
density of SO <sub>2</sub> (kg/m <sup>3</sup> )	2.668	2.668
chemical concentration mg/m <sup>3</sup>	13340	13340
PPM v	5000	5000
Humidity Flow (L/min)	0.34	1.84
diluent flow (l/min)	1.590	0.090
desired concentration (mg/m <sup>3</sup> )	1621	1621
Total H <sub>2</sub> O Flow (l/min)	0.011	0.058
SO <sub>2</sub> mass (mg)	320.5602	320.5602
mg SO <sub>2</sub> / g Carbon	40.1	40.1
Ct exposure (mg min/m <sup>3</sup> )	145908	145908
(a) H <sub>2</sub> O Volume (L)	0.96	5.18

**Figure 8** Saturator design (a) calculation for the required amount of water, (b) design diagram provided by manufacturer.

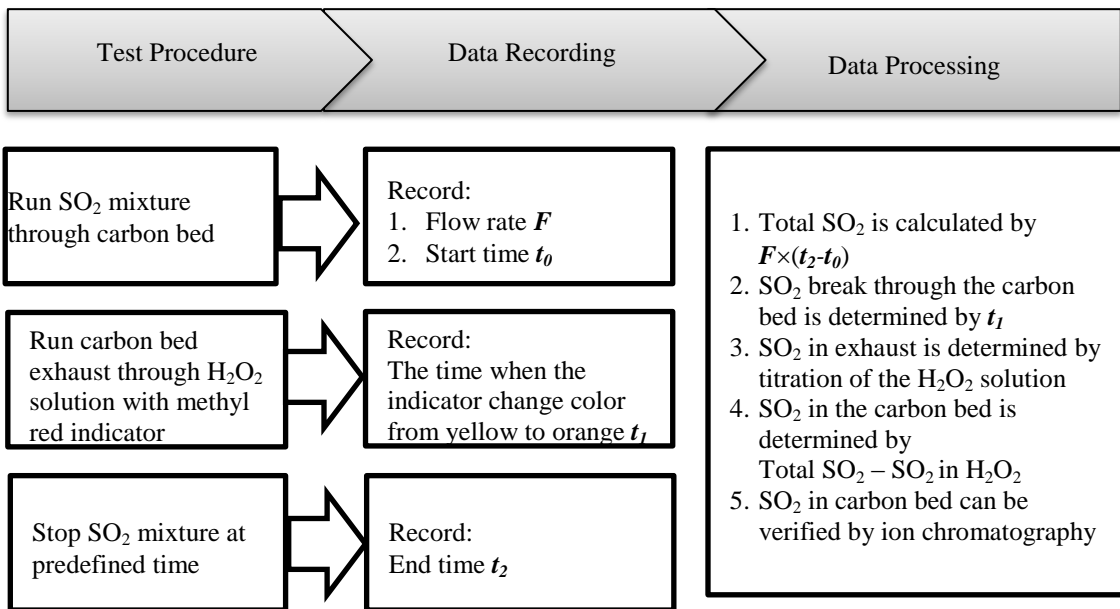
The saturator is pressure rated, and has a built-in pressure release valve. This feature is necessary to protect the testers if a pressure level exceeds the saturator pressure rating experienced during the tests. The saturator is expected to arrive in the beginning of the next reporting period, and we will test it with various humidity levels required by the brass board test scenarios.

During the process of building the testing apparatus, we performed SO<sub>2</sub> titration experiments to evaluate the absorption rate of SO<sub>2</sub> mixture in the H<sub>2</sub>O<sub>2</sub> solution. Several experiments were carried out on the partially built apparatus. The experimental setup is shown in Figure 7 (b). During the experiments, SO<sub>2</sub> mixture (0.5% SO<sub>2</sub>+99.5%

N<sub>2</sub>) run through the apparatus at a preset pressure level, and is absorbed by H<sub>2</sub>O<sub>2</sub> solution at the exhaust. The duration of running the SO<sub>2</sub> is controlled by the tester, and is used to control the amount of SO<sub>2</sub> getting into the H<sub>2</sub>O<sub>2</sub> solution. The pH level of the H<sub>2</sub>O<sub>2</sub> solution is measured by a pH meter. Titration procedure was used to evaluate the SO<sub>2</sub> contents in the H<sub>2</sub>O<sub>2</sub> solution. We have just started this testing procedure, and only very preliminary test data are available. We will conduct a few more experiments from the beginning of next reporting period, and report the data with data analysis results.

### 2.2.2 Suggestion on Ion chromatography testing procedure

We consulted with the private labs on precision measurement of different chemical detection methods that can be utilized for the qualification of SO<sub>2</sub> absorbed in the carbon bed. Through discussion, we found that ion chromatography can be utilized to assist detection and quantification of SO<sub>2</sub> in the carbon bed.



**Figure 9 Test procedures, data recording, and data processing schematic for the SO<sub>2</sub> test.**

We contacted chemical advisors in the chemical engineering department at USC, to aid in the team's SO<sub>2</sub> measurement. Through our continuous discussion, we found it was best to subject our experimental data with an ion chromatography to further solidify our detection practices SO<sub>2</sub> in the carbon bed sample. A brief schematic of test procedure, data recording and data processing for the SO<sub>2</sub> test is shown in Figure 9. A possible approach is to use the ion chromatography method to measure the SO<sub>2</sub> concentration in the carbon bed sample, and use the data to verify the data calculated from the titration method.

We have targeted two chemical labs which have the ion chromatography system for analysis of future test samples. One lab is located in the civil engineering department of USC, and the other lab is a local company (Shealy Environmental Labs) close to USC. In the next reporting period, we will discuss with the ECBC specialists about the possibility of using the ion chromatography method to assist the evaluation of SO<sub>2</sub> contamination level in the carbon bed. If approved, we will try to use the ion chromatography to detect the SO<sub>2</sub> contents in the carbon bed sample after contamination test.

## 3 PLANNED ACTIVITIES FOR NEXT QUARTER (JANUARY-MARCH 2014)

For the upcoming quarter (January-March), we will continue work on building the new carbon bed SO<sub>2</sub> contamination apparatus (Task 4), and on: Specifications for prototype development and final report (Task 5). We will report to ECBC specialists the progress of the tasks, and discuss the brass board design draft. Once the design gets approval, we will start constructing the brass board tester.

### Perform Task 4: EMIS-ECIS bench top demonstrator design and construction

This task will focus on designing and constructing an integrated bench top experimental setup that will demonstrate the different aspects of the EMIS-ECIS methodology. We will use the design of specialized experiments from Task

2, extend them, and then assemble them into a multifunction platform. The design will be adapted to the model development. A breadboard system will be developed for the demonstrator.

**Perform Task 5: Specifications for prototype development and final report**

In this task, we will develop specifications for a prototype to transition the research results and fully implement them into an actual piece of equipment. As we mentioned earlier, the focus of the present project is to understand the PWAS-carbon bed impedance model; full development of residual life estimation system will need further efforts. The methodology developed in this project should be transitioned to practice and implemented into a proof of concept prototype to address the issues Army CBR Filtration concern. We will provide the specifications of such a prototype, to fully demonstrate the capacity of proposed methodology, and pave the way to the next step of development. In this task, we will also compile the comprehensive final report of the project.

**4 REFERENCES**

- [1] Farrar, C. R, Duffey, T. A.; Doebling, S. W.; Nix, D. A. (1999) “A Statistical Pattern Recognition Paradigm for Vibration-Based Structural Health Monitoring”, Proceedings of the 2nd International Workshop on Structural Health Monitoring, September 8-10, 1999, Stanford University, Stanford, CA, USA, pp. 764-773
- [2] Fritzen; C. P.; and Bohle, K. (1999) “Parameter Selection Strategies in Model-Based Damage Detection”, *Proceedings of the 2<sup>nd</sup> International Workshop on Structural Health Monitoring*, September 8-10, 1999, Stanford University, Stanford, CA, USA, pp. 901-911
- [3] Farrar, R. C.; Sohn, H.; Fugate, M. L, Czarnecki, J. J (2001) “Statistical Process Control and Projection Techniques for Damage Detection”, Proceedings of the SPIE’s Conference on Advanced Nondestructive Evaluation for Structural and Biological Health Monitoring, March 4-8, 2001, Newport Beach, CA, USA, Vol. 4335, pp. 1-19

# **Quarterly REPORT**

**For the period April - June 2014**

## **PWAS EMIS-ECIS Active Carbon Filter Residual Life Estimation Methodology**

Submitted by:

Victor Giurgiutiu and Jingjing Bao  
University of South Carolina  
300 Main Street, Columbia SC 29208  
Telephone: (803) 777-8018  
Email: victorg@sc.edu

Submitted to:

Gregory W. Peterson  
CBR Filtration Branch  
U. S. Army Edgewood Chemical Biological Center  
5183 Blackhawk Road, Bldg. 3549  
APG, MD 21010  
Telephone: (410) 436-9794  
Email: gregory.w.peterson@us.army.mil

Date June 30, 2014

Project Number: W911NF\_11\_1

Capability Area: CB filtration



## Contents

1	Project and report overview.....	2
2	Progress during the reporting period (January-March 2013).....	3
2.1	Improving the PWAS EMIS tester design, the testing procedure, and the EMIS data analysis method.....	3
2.2	Experimental validation of the PWAS EMIS tester setup and the data analysis method.....	9
3	Planned activities for next quarter (July-September 2014).....	10

### 1 PROJECT AND REPORT OVERVIEW

In this reporting period, we focused on two major activities: (a) improving the PWAS EMIS tester design, the test procedure, and (b) experimental validation of the PWAS EMIS tester setup and the data analysis method. At the end of this reporting period, we have finalized the EMIS tester setup design and construction. We have performed multiple experiments to validate the PWAS EMIS testing procedure. The data analysis method was examined with the experimental data.

#### **Improving the PWAS EMIS tester design, the testing procedure, and the EMIS data analysis method**

We have integrated the mass flow controllers into the carbon bed contamination setup. The mass flow controllers are able to control the flow of  $N_2$  and  $SO_2$  into the carbon bed. We have performed several experiments to adjust the testing setup. An important modification to the system is to include a purge line from the  $N_2$  cylinder to the  $SO_2$  route to flush the  $SO_2$  mass flow controller after experiments. By reviewing the data from multiple experiments, improvement of the experimental procedures are made: such as reducing the frequency range and increasing the frequency resolution when collecting the impedance data. We have adjusted the frequency range to cover only the first PWAS resonance peak (200 kHz-400 kHz), and increase the frequency resolution (0.1 kHz) to provide better peak quality for the analysis.

The mechanical characters of the carbon bed change slowly during the experiment. Hence, the PWAS EMIS spectrums often have only very small variations between samples. A data analysis method is experimented to capture the trend of change in the EMIS spectrum. This method is similar to the full width at half maximum (FWHM) method implemented in the fiber Bragg grating (FBG), where the intensity of the light reflected by the FBG at the half peak frequency is used to detect the strain change in the FBG. A brief list of the steps is:

1. the frequency of the data point at half amplitude of the resonance peak is found from the baseline data
2. the impedance value is extracted from each test data set at the frequency determined in step 1
3. the impedance value extracted from step 2 is compared with the testing parameter such as exposure time or temperature

#### **Experimental validation of the PWAS EMIS tester setup and the data analysis method**

Multiple experiments are carried out to validate the PWAS EMIS tester setup and procedures. The data analysis method is verified with the experimental data. The experimental data and data analysis plots are presented in the appendix section. From the experimental, some important observations are:

1. The impedance and admittance resonance peaks show higher frequency and amplitude with the progression of the experiment, as shown by the LHP and RHP plot is all the appendix plots.
2. Preliminary data analysis shows that the PWAS impedance is affected by the temperature in the carbon bed, as shown by *Appendix 5: Temperature test, Jun 18* on page 20.
3. The introduction of small amount of  $SO_2$  (approximately 0.13% by volume) has noticeable effects on the impedance peak, as shown by *Appendix 7:  $N_2$  test+ $SO_2$  test, Jun 19* on page 24
4. More data analysis is needed to differentiate the effect of pure temperature change,  $N_2$  flow, moisture level, and the  $SO_2$  contamination.

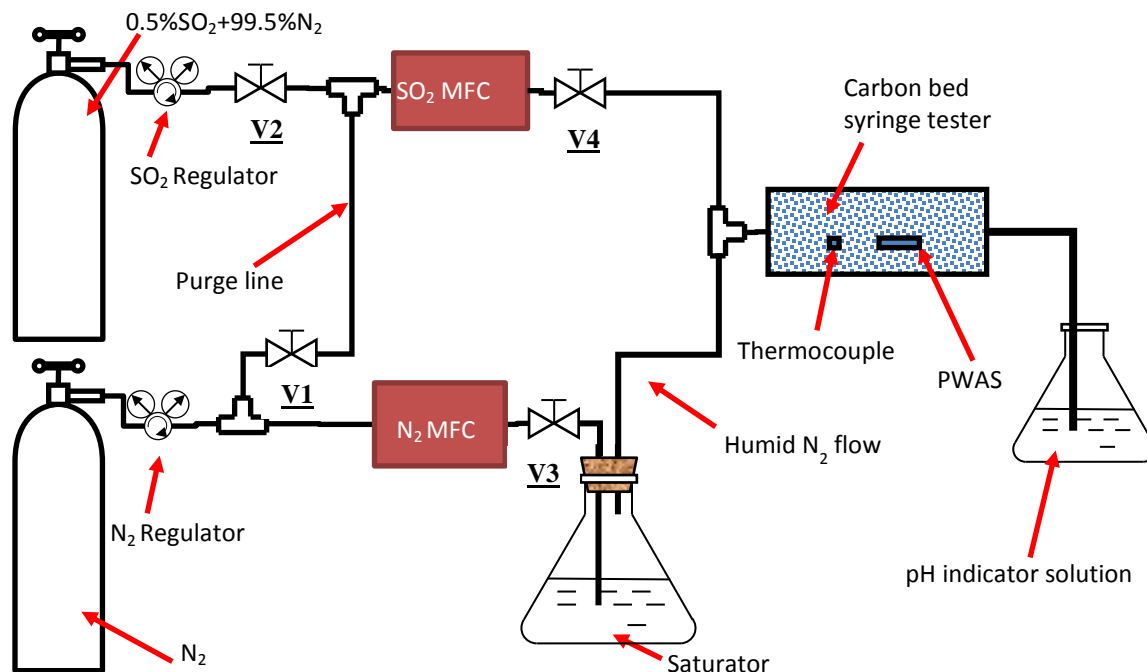
The progress details are covered in the following sections.

## 2 PROGRESS DURING THE REPORTING PERIOD (JANUARY-MARCH 2013)

### 2.1 Improving the PWAS EMIS tester design, the testing procedure, and the EMIS data analysis method Improving the tester design

In this reporting period, we improve the PWAS EMIS tester design, to provide precise control of the SO<sub>2</sub> and N<sub>2</sub> flows. Figure 1 shows the updated test design sketch. We have integrated the mass flow controller (MFC) into the carbon bed contamination setup. Two MFCs are able to control the flow of N<sub>2</sub> and SO<sub>2</sub> into the carbon bed. The MFCs are purchase from Sierra Instruments, model SmartTrak 50 series, with full scale flow rate of 5 SLPM, and accuracy of 1.0% of the full scale. The flow rate reading and control is through a RS-232 interface. A control program runs on a PC to monitor and control the flow rate remotely.

An important modification to the system is to include a purge line from the N<sub>2</sub> cylinder to the SO<sub>2</sub> route to flush the SO<sub>2</sub> MFC after experiments. As shown in Figure 1, the purge line is controlled by the V1 valve. The purge procedure is described in the next section.



**Figure 1 PWAS EMIS tester design sketch.**

#### Testing procedure

After improving the PWAS EMIS tester design, we modify the testing procedure to adapt to the improved design. As we introduced several cut-off valves to prevent moisture back flow and to purge the MFC, the operating procedure now includes more steps to perform an experiment. A typical experimental procedure is described below.

#### *Indicator Solution*

1. Pour 500mL of deionized water into a beaker.
2. Add 5 drops (0.25 mL) of sodium hydroxide to the water.
3. Add 5 drops (0.25 mL) of phenolphthalein to the solution.
4. Mix by swirling the beaker.

### *Loading the Carbon Bed Syringe Tester*

1. Pour 20 grams of carbon granules into the open section of the syringe. Shake the carbon granules to make room for the PWAS to move into the carbon bed.
2. Insert the plunger into the syringe. Move the PWAS and thermocouple into the carbon granules. Shake the carbon granules to cover the PWAS and thermocouple completely.
3. Press the plunger to form a tight seal with the back of the syringe.
4. Check the PWAS impedance and thermocouple readings to verify the electrical connections.

### *Initiating AND Controlling Gas Flow (See Diagram for References)*

1. Begin the process by setting the MFCs to their specified flow rates (Adjustments) for each gas. (N<sub>2</sub>: 1.93 SLPM and SO<sub>2</sub>: 0.677 SLPM for this experiment)
2. At this point V1, V2, V3, and V4 should all be closed. With V1 closed, the N<sub>2</sub> gas will follow the path to N<sub>2</sub> MFC.
3. Open the pressure valve on the top of the N<sub>2</sub> tank.
4. Open V3.
5. Turn the regulator valve on the N<sub>2</sub> regulator to allow the gas to flow. You will know the gas is flowing, because the control system will show a flow rate and bubbles will be produced in the indicator solution.
6. Open the pressure valve on the top of the SO<sub>2</sub> tank.
7. Open V2 and V4.
8. At this point gas should be flowing through both portions of the system. This will be shown on the control system screen. The control windows for both gases should show a flow rate equal to the specified flow rate. (There will be fluctuation at the beginning of the process; this is an equilibration period to expend the excess pressure that had built up.)

### *Experiments and Measurements*

1. Prior to running each experiment, a pH measurement of the indicator solution should be taken and recorded. A pH meter should be placed into the indicator solution and the initial pH measurement should be recorded. After the experiment is completed, the final pH of the indicator solution should be measured. (If several experimental processes are being run consecutively, a pH measurement should be taken after each individual experiment.)
2. The primary measurements that are to be studied are the change in impedance and the temperature inside the syringe. One measurement for each of these variables should be taken at consecutive 1 minute intervals over the course of the experiment. These measurements should be recorded and analyzed.

### *Shutting Down and Purging the System*

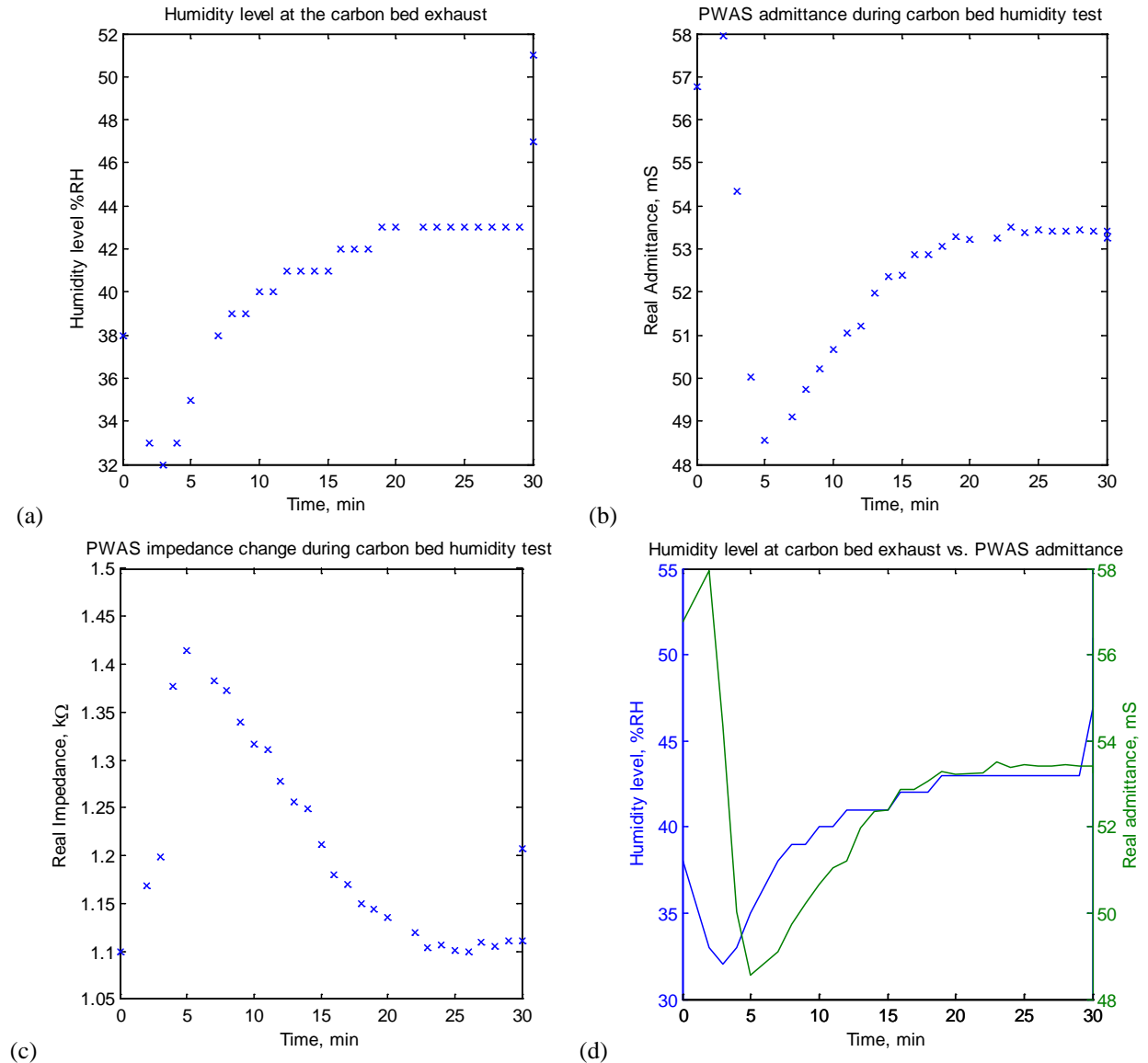
To avoid any backflow of humidified N<sub>2</sub> or any residual SO<sub>2</sub>, which may damage the MFCs, a specific protocol is followed to shut down the system and to purge the SO<sub>2</sub> MFC.

1. First the flow of SO<sub>2</sub> must be halted. To do this, close the pressure valve on the top of the SO<sub>2</sub> tank.
2. Wait until the gauges on the top of the tank will both have fallen to 0 psi, then close V<sub>2</sub>.
3. Close V3 and open V1, so that the N<sub>2</sub> will flow along the path to the SO<sub>2</sub> MFC.
4. Change the setting on the SO<sub>2</sub> MFC's Adjustment to 2 SLPM.
5. Allow the gas to run for several minutes (5 min. was used in this test).
6. After the gas has been allowed to run, close the pressure valve on the N<sub>2</sub> tank.
7. Once the control system shows no flow and the gauges on the N<sub>2</sub> tank both show 0 psi, close V4.
8. Close V1, so that the N<sub>2</sub> will follow the path to the N<sub>2</sub> controller.

### EMIS data analysis method

The PWAS electrical impedance (or admittance) spectrum peaks identify the anti-resonance (or resonance) modes of the PWAS transducer. Once attached to a structure, the PWAS electrical impedance couples with the mechanical impedance of the structure. Hence, the EMIS method is able to identify the structural impedance change by the PWAS impedance spectrum. The frequency shift and amplitude change of the impedance peaks are two of the important indicators for the EMIS method.

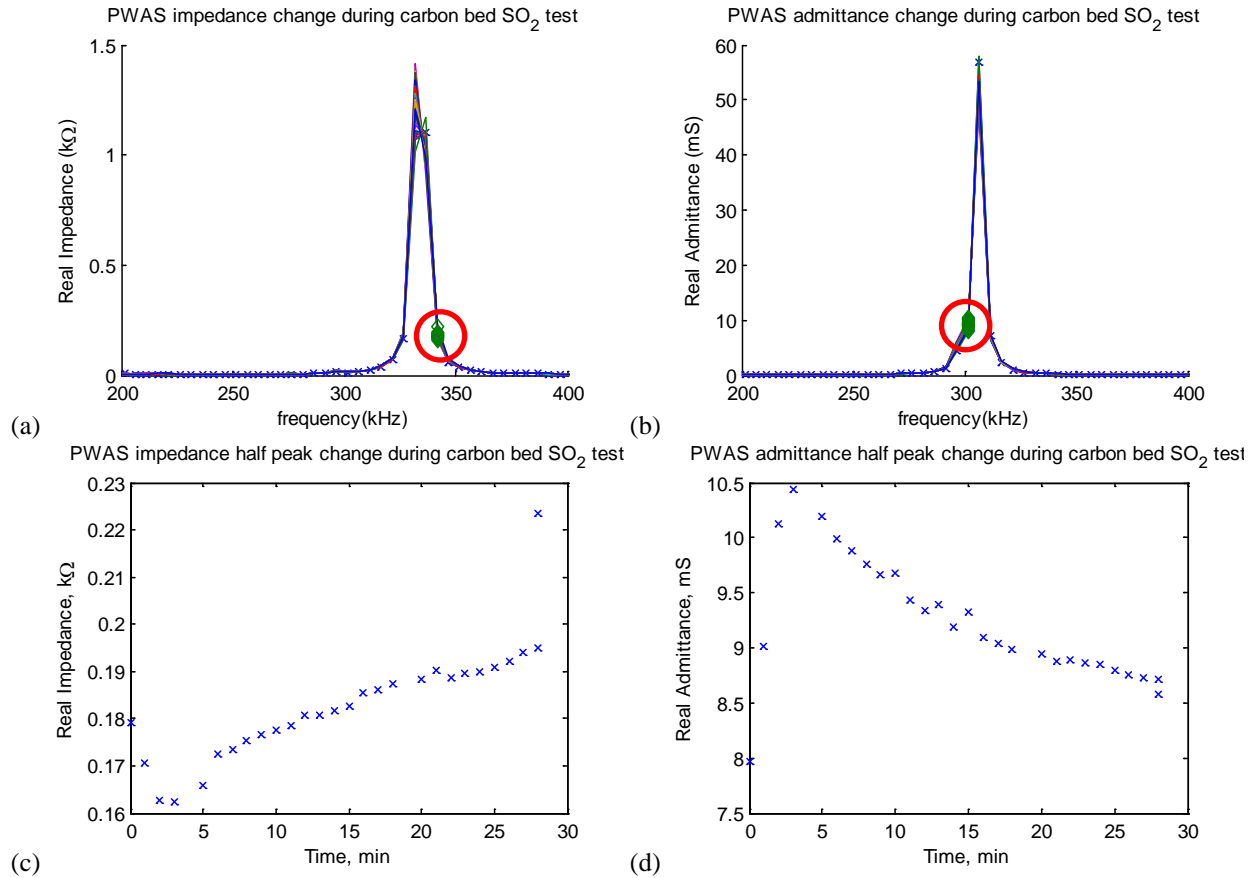
In this project, PWAS transducers are embedded into the carbon bed, and monitor the local mechanical impedance change introduced by  $\text{SO}_2$ , water vapor, and other contaminants. The experimental data from the previous several reporting periods show the connection between the PWAS impedance peak and the carbon bed conditions, such as the carbon bed compact pressure, the humidity level, and the temperature. In the previous reporting period, the data from carbon bed humidity test was reported, as shown in Figure 2. The data suggests that the PWAS admittance peak amplitude is correlated to the humidity level of the carbon bed.



**Figure 2** Experimental data from the humidity test on carbon bed embedded with PWAS. (a) Humidity level measured at the carbon bed exhaust, (b) the PWAS admittance peak value during the experiment, (c) the PWAS impedance peak value during the experiment, and (d) the comparison between the humidity level at carbon bed exhaust and the PWAS admittance peak value.

*Reduce the EMIS frequency range and increase frequency resolution*

In this reporting period, we review the data set to examine the data analysis method. The impedance and admittance resonance peak was extracted and plotted in Figure 3 (a) and (b). the 'x' marked plots are baseline data. It can be noticed that the data points are very coarse at the peak. There are actually only two points on the top of the impedance and only one for the admittance plot. It is also noticed that all the impedance data seems overlapping with each other, which makes the peak value trend of change questionable. The reason we chose the coarse frequency resolution was to reduce the data collection time in order to capture the impedance every minute during the experiment. It is also because the frequency range was relative wide to include from 1 kHz to 5 MHz, such we can monitor the PWAS behavior in wider frequency band. After reviewing this data set, we redesign the testing procedure to reduce the frequency range to 200 kHz – 400 kHz, and increase the frequency resolution to 0.1 kHz/point.

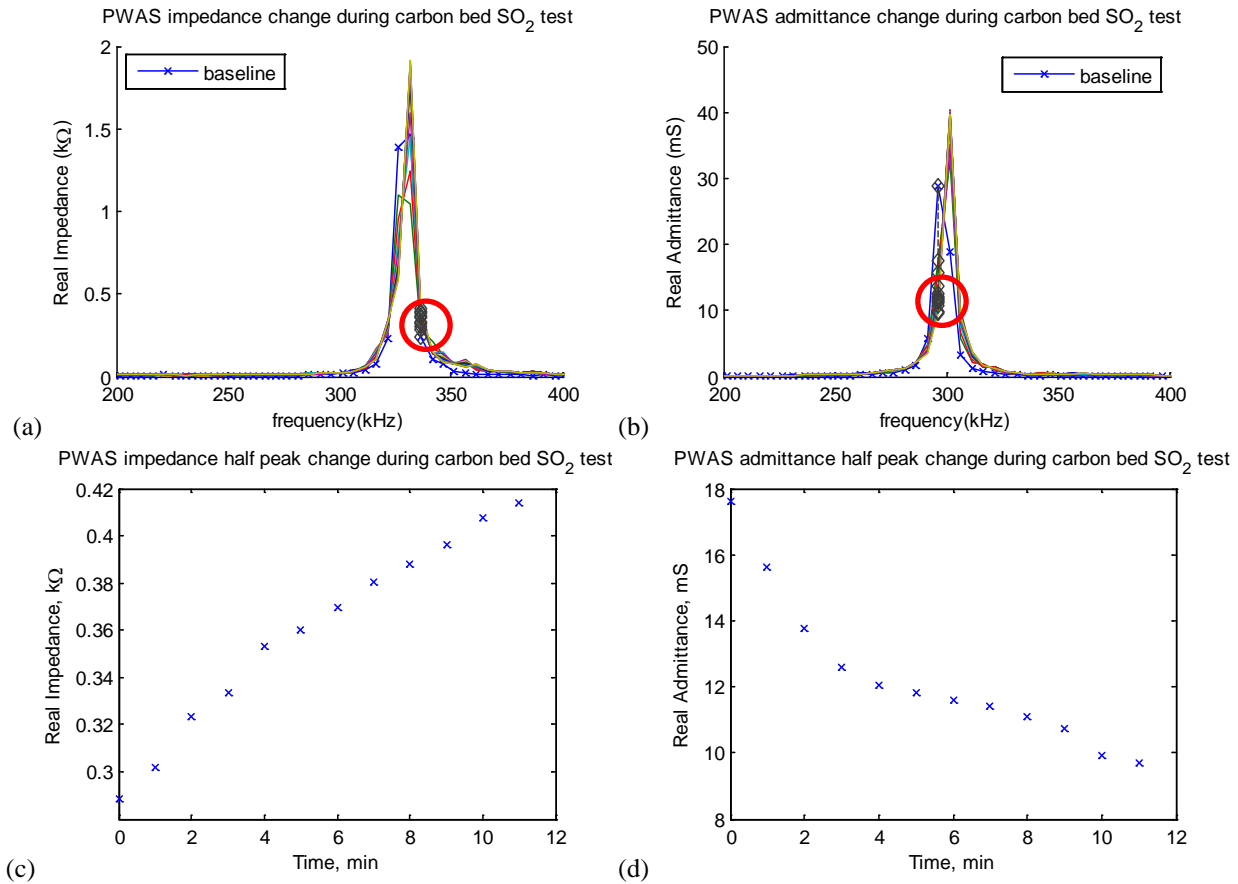


**Figure 3 Review the experimental data from the humidity test on carbon bed embedded with PWAS: (a) impedance spectrum at the resonance peak, (b) admittance spectrum at the resonance peak, (c) impedance change history at the half maximum frequency, and (d) admittance change history at the half maximum frequency. The makers on (a) and (b) show the data points at the half maximum frequency (3/28/2014 data)**

Another important improvement is to modify the data analysis method. Because the mechanical characters of the carbon bed change slowly during the experiment, the PWAS EMIS spectrums often have only very small variations between samples. A data analysis method is experimented to capture the trend of change in the EMIS spectrum. A brief list of the steps is:

1. the frequency of the data point at half amplitude of the resonance peak is found from the baseline data
2. the impedance value is extracted from each test data set at the frequency determined in step 1
3. the impedance value extracted from step 2 is compared with the testing parameter such as exposure time or temperature

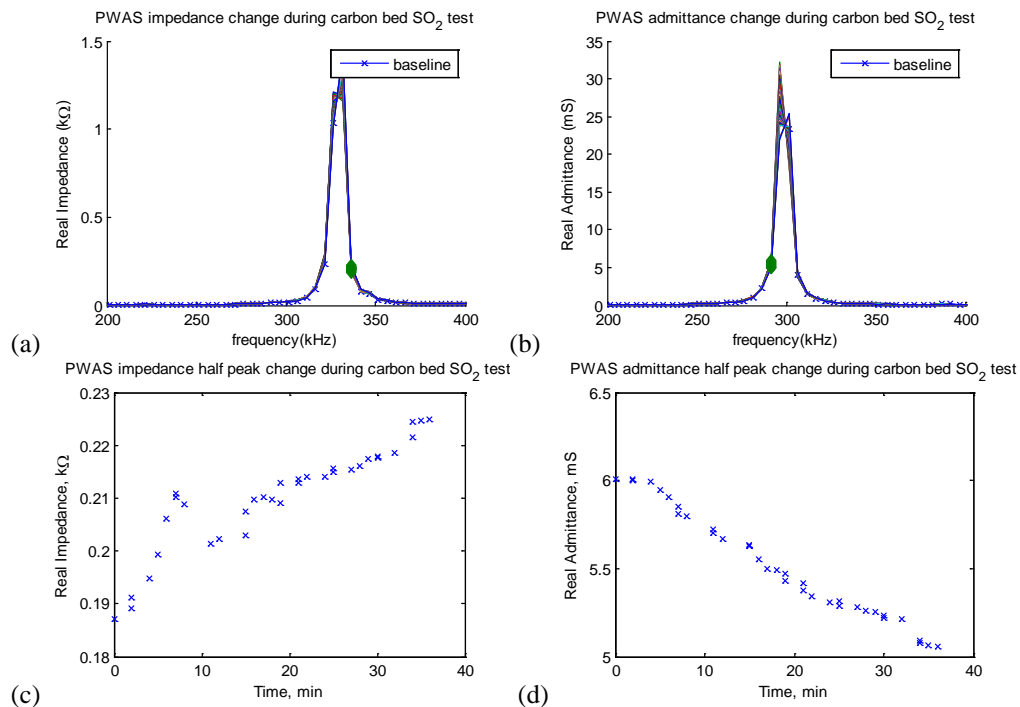
This method is similar to the full width at half maximum (FWHM) method implemented in the fiber Bragg grating (FBG), where the intensity of the light reflected by the FBG at the half peak frequency is used to detect the strain change in the FBG[1]. Figure 3 (a) and (b) show with markers the actual points collected from the impedance and admittance spectrum at the half maximum frequency. Figure 3 (c) and (d) show the collected data points plot against the experimental time, which indicates the correlation between the impedance/admittance and the humidity level of the carbon bed. More experiments are carried out during this reporting period, and the data analysis method is applied to verify the results.



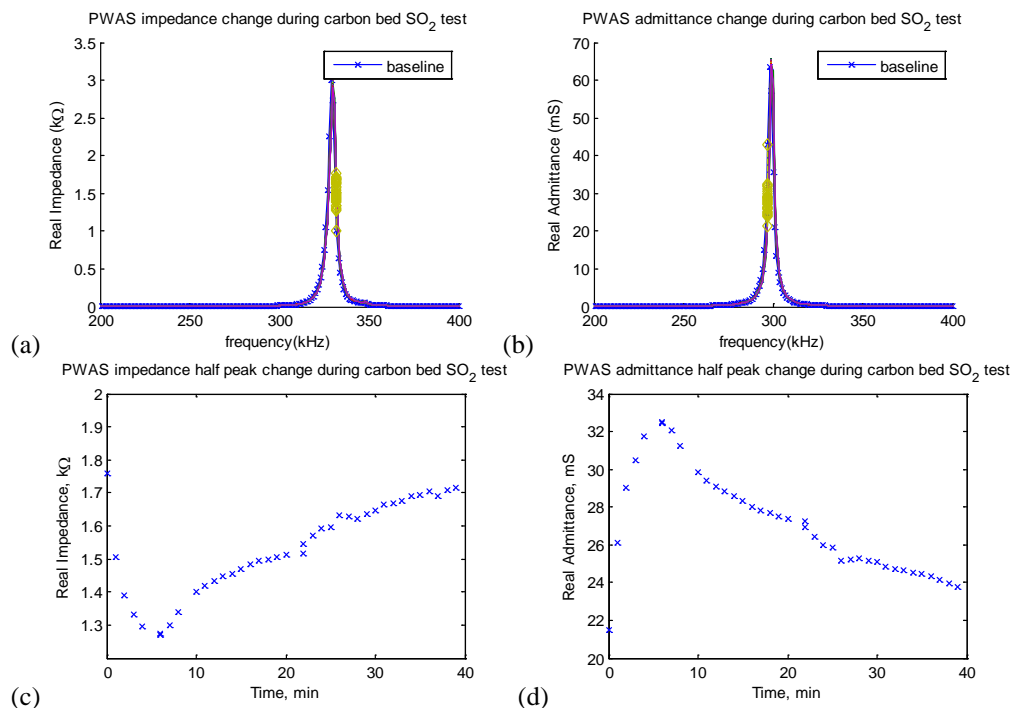
**Figure 4 PWAS impedance data from the humidified N<sub>2</sub> test on carbon bed (a) impedance resonance peak, (b) admittance resonance peak, (c) impedance change history at the half maximum frequency, and (d) admittance change history at the half maximum frequency. (4/7/2014 data)**

Figure 4 shows another test of carbon bed contamination with humidified N<sub>2</sub>. The frequency resolution is the same as the previous test. The spectrum plots (a) and (b) again shows poor frequency resolution. If we compare the peak amplitude plots in (c) and (d) with Figure 2(c) and (b), the differences are clear: in Figure 4 (c), the impedance increases with time, while in Figure 2(c), the impedance decreases with time. Then we apply the half maximum frequency method, and the results are shown in Figure 4 (e) and (f). We can identify that the trend of change matches Figure 3 (c) and (d).

Figure 5 and Figure 6 show the first group of carbon bed contamination test with SO<sub>2</sub> and humidified N<sub>2</sub>. The data in Figure 5 is again with poor frequency resolution, while the data in Figure 6 is improved with 1kHz frequency resolution. Same steps are taken to process the experimental data. We can notice the similarity of the trend of change of the impedance/admittance at the half maximum frequency, despite the variation in the trend of spectrum peak amplitude in Figure 6 (c).



**Figure 5** PWAS impedance data from the carbon bed  $\text{SO}_2$  and humidified  $\text{N}_2$  test (a) impedance resonance peak, (b) admittance resonance peak, (c) impedance change history at the half maximum frequency, and (d) admittance change history at the half maximum frequency (4/24/2014 data)



**Figure 6** PWAS impedance data from the  $\text{SO}_2$  and humidified  $\text{N}_2$  test (a) impedance resonance peak, (b) admittance resonance peak, (c) impedance change history at the half maximum frequency, and (d) admittance change history at the half maximum frequency. (4/30/2014 data)

With the preliminary result, we can see that the half maximum frequency method seem to be more consistent and robust than the peak amplitude method. More experiments still needed to validate and verify this method.

## **2.2 Experimental validation of the PWAS EMIS tester setup and the data analysis method**

In this reporting period, we have performed multiple carbon bed contamination experiments with embedded PWAS transducers. The nitrogen flow is humidified by a saturator, and then mixed with the sulfur dioxide (0.5% by volume in nitrogen). The exhausts of the carbon bed run through the hydrogen peroxide solution to capture the sulfur dioxide breakthrough.

### **Experiment set 1, May 29 – June 3**

Two sets of experiments are carried out.

1. Nitrogen is 2.2 L/m
2. Nitrogen is 1.930 L/min, and 0.5% SO<sub>2</sub>+99.5% N<sub>2</sub> is 0.27 L/min

This is designed to show the effects of the SO<sub>2</sub> contamination. Each experiment runs 30 minutes. The PWAS impedance spectrum and the carbon bed temperature are recorded at one minute intervals.

The experimental data are analyzed and presented in the attached pages. The parameters presented are:

1. The impedance resonance peaks
2. The admittance resonance peaks
3. The impedance peak amplitude and frequency change vs. the carbon bed temperature
4. The admittance peak amplitude and frequency change vs. the carbon bed temperature
5. The impedance half peak amplitude change vs. time during the experiment
6. The admittance half peak amplitude change vs. time during the experiment

### **Experiment set 2, May 18 – June 19**

Over the days of June 18th and June 19th, four separate experiments were conducted. In previous experiments, it was observed that the temperature seemed to have some correlation with the change in the impedance. It was also observed that, on hot days, the temperature inside the fume hood decreased when the vent was turned on in comparison to when the vent was off. To analyze the effect this decrease in temperature produced, four individual experiments were conducted, and the measurements were analyzed.

On the date of June 18th, two experiments were run consecutively. The first experiment was to analyze the effect the vent had on the temperature inside the carbon bed. The system was set up as would normally be done for any test, but no gas was run. The experiment began with the vent off, and baseline measurements of temperature and impedance were taken. The fan was then turned on, and measurements were taken at 1 minute intervals for 30 minutes. Over the course, record shows a starting temperature of 23.3 °C and an ending temperature of 21.6 °C.

Following the collection of data for the temperature only experiment, an experiment in which both the N<sub>2</sub> and SO<sub>2</sub> gasses were run was performed. The gasses were allowed to flow over the carbon bed, in accordance with the previously stated protocols. These gasses were allowed to run for 40 minutes, and temperature and impedance measurements were taken at 1 minute intervals. During this experiment, there was a great increase in temperature. After just 1 minute of gas flow, the temperature inside the carbon bed had risen from 21.6 °C to 25.8 °C. At the 2 minute mark, the temperature had reached 27.2 °C. After this time the temperature seemed to lower and then level off, with an average temperature of 26.9 °C.

To further examine the effect that the change in temperature had on the impedance, two consecutive experiments were performed on June 19th. Prior to these experiments, the vent fan was allowed to run in an attempt to allow the temperature under the hood to equilibrate. In the first experiment, only the humidified N<sub>2</sub> was run through the system. The gas was allowed to run for 30 minutes and temperature and impedance measurements were taken at 1 minute intervals. Once again, an increase in temperature was initially observed, with the temperature rising from 26.4 °C at the 0 minute mark, and reaching 30.9 °C by the 5 minute mark. The subsequent minutes once again showed a slight decrease, followed by a level period that had an average temperature of 29 °C.

After the humidified N<sub>2</sub> experiment, the gas flow was then stopped, to allow for a pH measurement to be obtained. In the time that the flow of gas was stopped, the temperature dropped substantially, from 28.3 °C to 23.2 °C. After



taking the measurement, both the N<sub>2</sub> and the SO<sub>2</sub> were run for 30 minute. Once again measurements of temperature and impedance were taken every minute. During this time a substantial rise in temperature was notice immediately. By the two minute mark, the temperature rose from 23.2 °CC to 27.6 °C. After this time, the temperature evened out and remained fairly consistent with an average temperature of 27.4 °C. In comparison, both experiments that involved both the N<sub>2</sub> and the SO<sub>2</sub> showed a similar trend of temperature change, but the temperatures were not identical.

The experimental data are analyzed and presented in the attached pages. The parameters presented are:

1. The impedance resonance peaks
2. The admittance resonance peaks
3. The impedance peak amplitude and frequency change vs. the carbon bed temperature
4. The admittance peak amplitude and frequency change vs. the carbon bed temperature
5. The impedance half peak amplitude change vs. time during the experiment
6. The admittance half peak amplitude change vs. time during the experiment

The experimental data and analysis plots are presented in the appendix section.

#### **The observations from the experimental data**

5. The impedance and admittance resonance peaks show higher frequency and amplitude with the progression of the experiment, as shown by the LHP and RHP plot is all the appendix plots.
6. Preliminary data analysis shows that the PWAS impedance is affected by the temperature in the carbon bed, as shown by *Appendix 5: Temperature test, Jun 18* on page 20.
7. The introduction of small amount of SO<sub>2</sub> (approximately 0.13% by volume) has noticeable effects on the impedance peak, as shown by *Appendix 7: N<sub>2</sub> test+SO<sub>2</sub> test, Jun 19* on page 24
8. More data analysis is needed to differentiate the effect of pure temperature change, N<sub>2</sub> flow, moisture level, and the SO<sub>2</sub> contamination.

#### **PLANNED ACTIVITIES FOR NEXT QUARTER (JULY-SEPTEMBER 2014)**

This project is approaching the end. Our focus for the next reporting period is to assemble the existing experimental data and analysis result, and finish the final project repot. For the upcoming quarter (July-September), we will use all the test results to improve the data analysis method. We will report to ECBC specialists the progress of the tasks, and discuss the final outcome of the research results. At end of the reporting period, we will complete and submit the final project report. We will also make ready any PWAS EMIS testing hardware, procedure, data and data analysis software required by the ECBC.

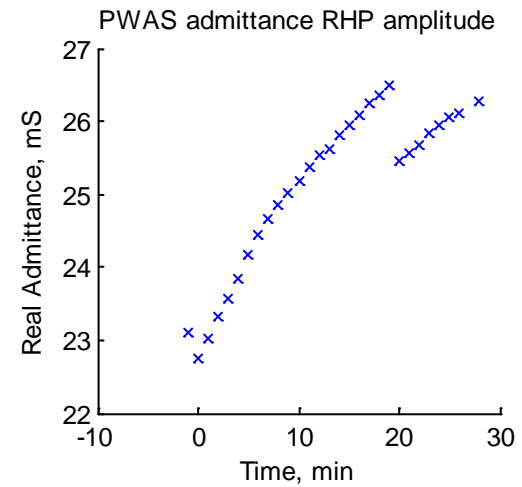
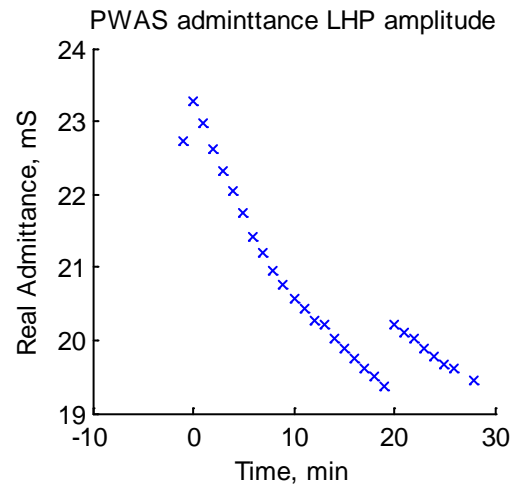
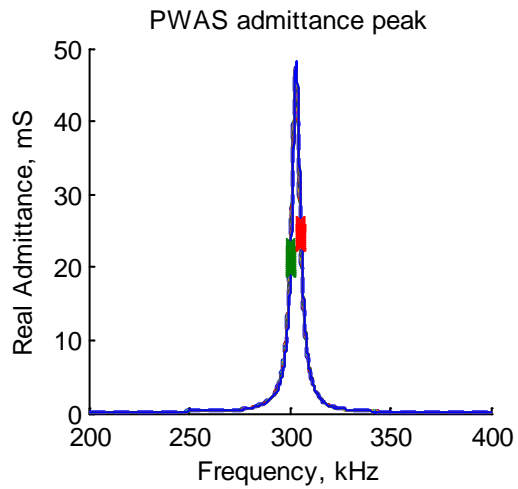
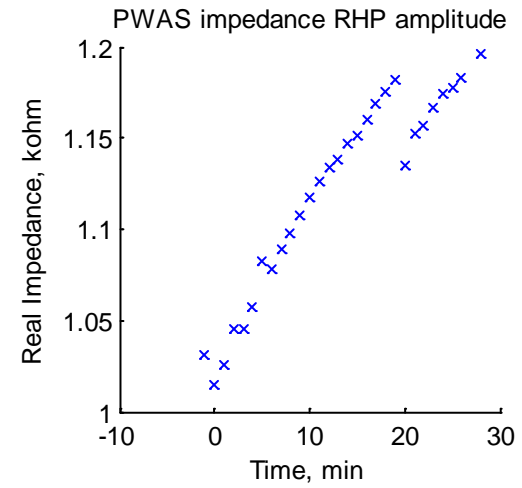
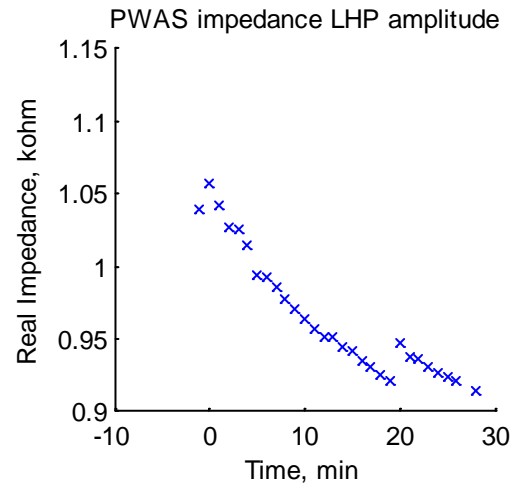
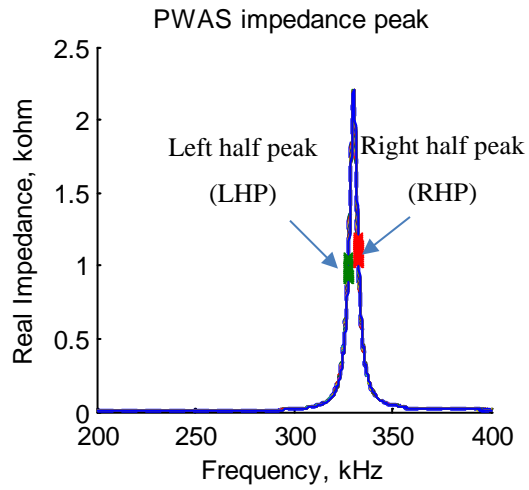
#### **References**

1. Lin, B.; Giurgiutiu, V. (2013) “**Exploration of Ultrasonic Guided Wave Detection with Optical Fiber Sensors and Piezoelectric Transducers**”, 9<sup>th</sup> *International Workshop on Structural Health Monitoring*, IWSHM, Sept. 10-12 2013, Stanford University, CA, pp. 1559-1566

### 3 APPENDIX: EXPERIMENTAL DATA AND ANALYSIS PLOTS

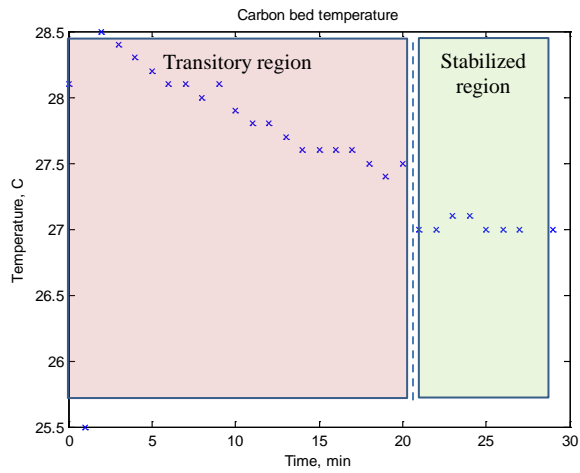
#### Experiment set 1, May 29 – June 3

Appendix 1: Nitrogen Only, May 30

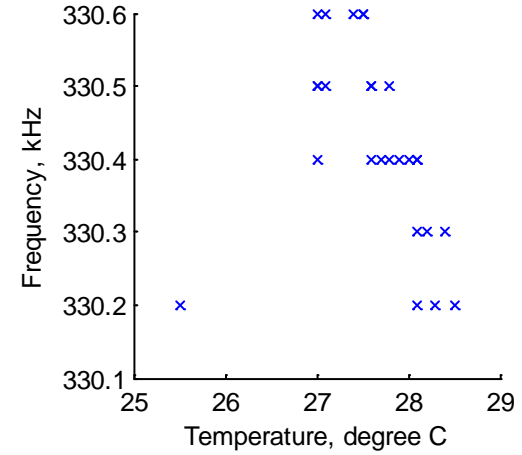
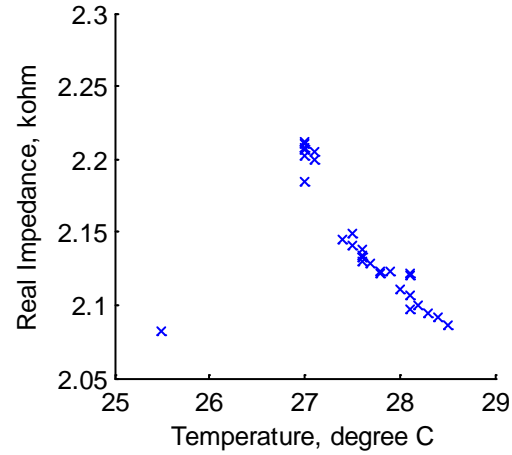


**Experiment set 1, May 29 – June 3**

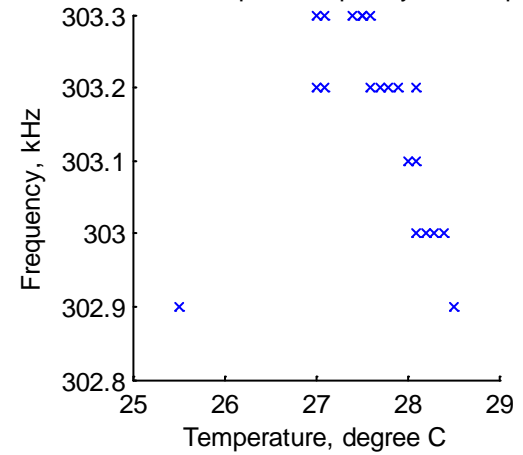
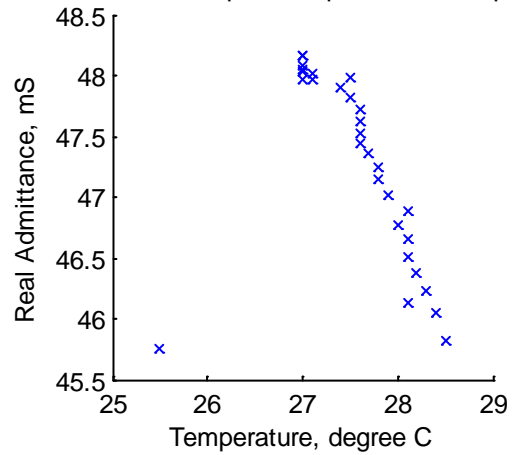
Appendix 1: Nitrogen Only, May 30 (cont.)



PWAS Impedance peak amplitude vs. temperat PWAS Impedance peak frequency vs. tempera

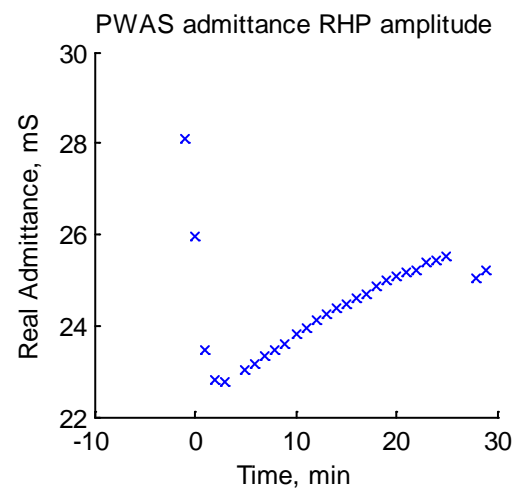
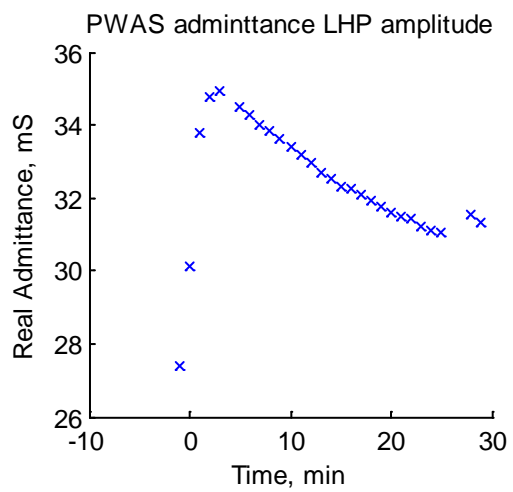
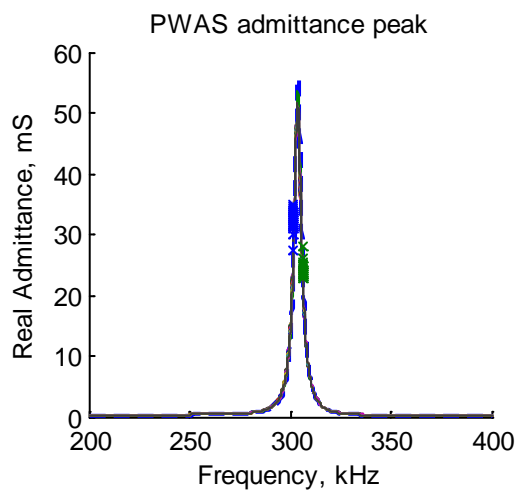
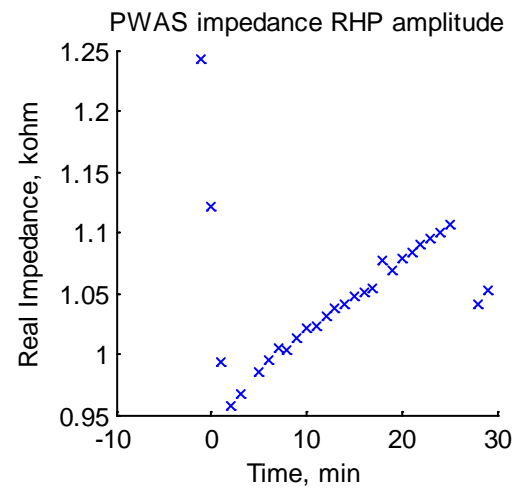
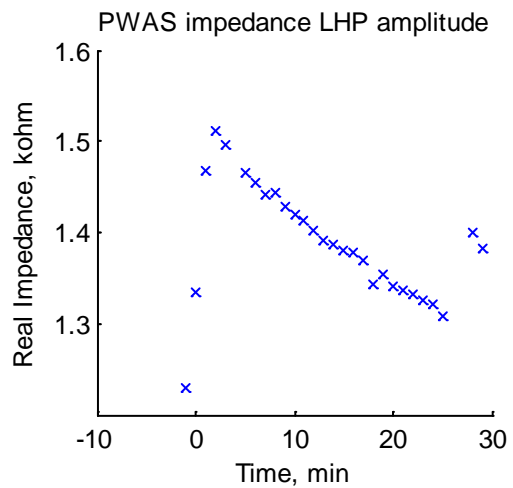
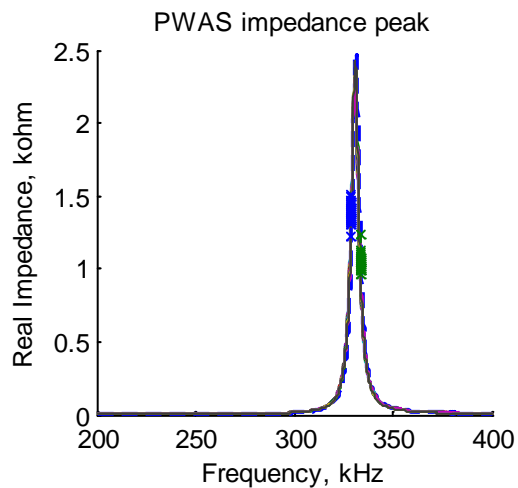


PWAS admittance peak amplitude vs. temperat PWAS admittance peak frequency vs. tempera



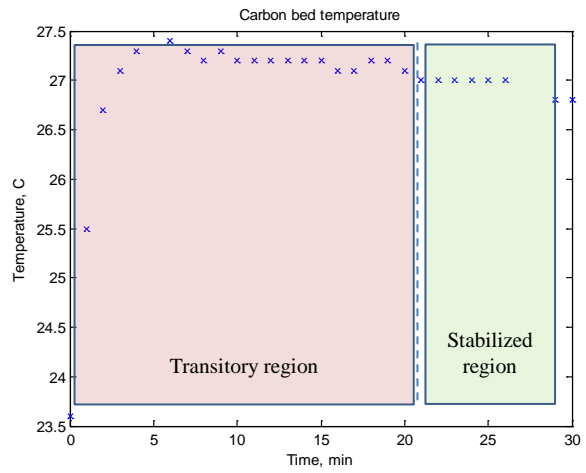
**Experiment set 1, May 29 – June 3**

Appendix 2: Nitrogen Only, June 3

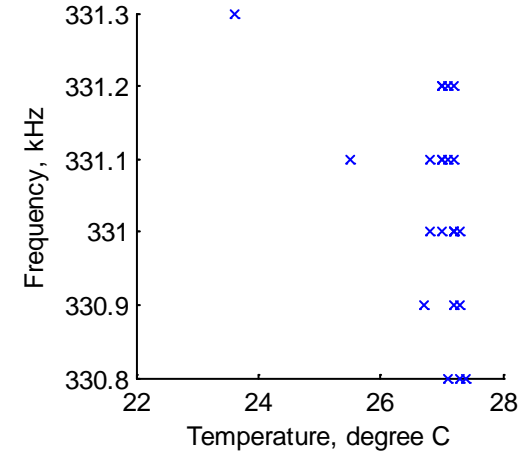
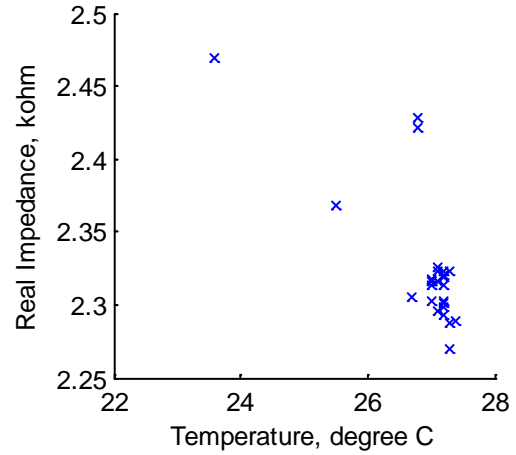


**Experiment set 1, May 29 – June 3**

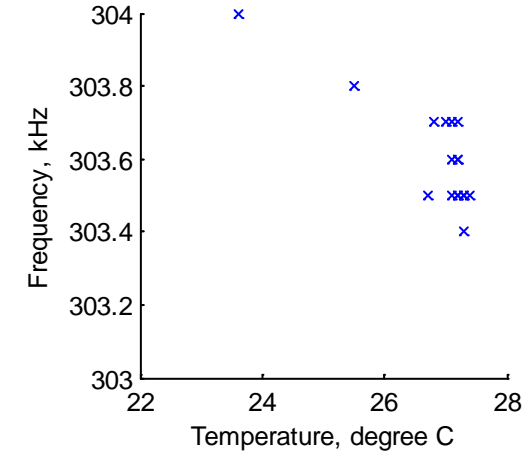
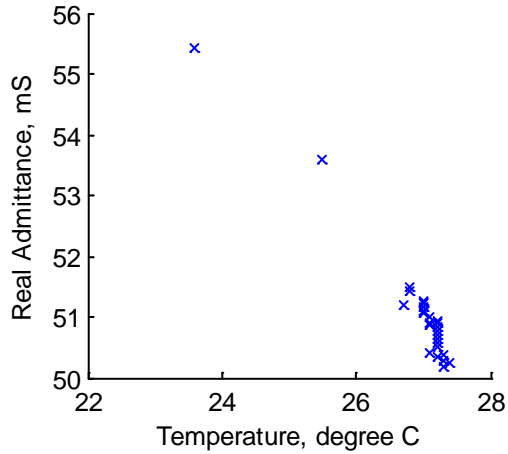
Appendix 2: Nitrogen Only, June 3 (cont.)



PWAS Impedance peak amplitude vs. temperature PWAS Impedance peak frequency vs. temperature

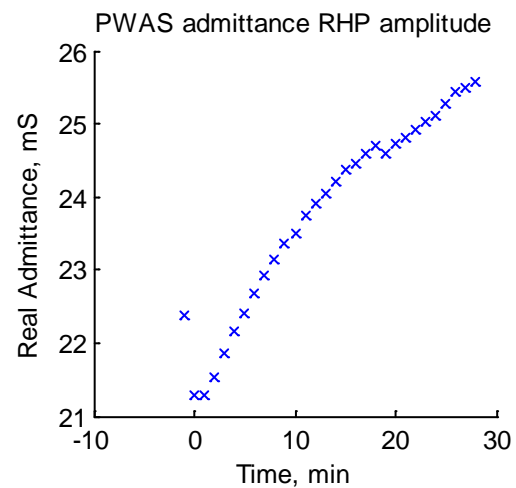
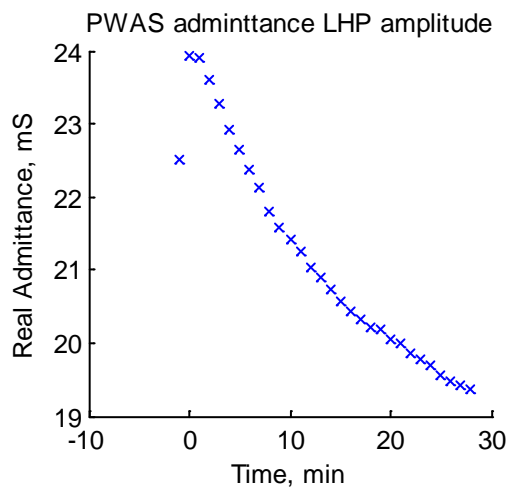
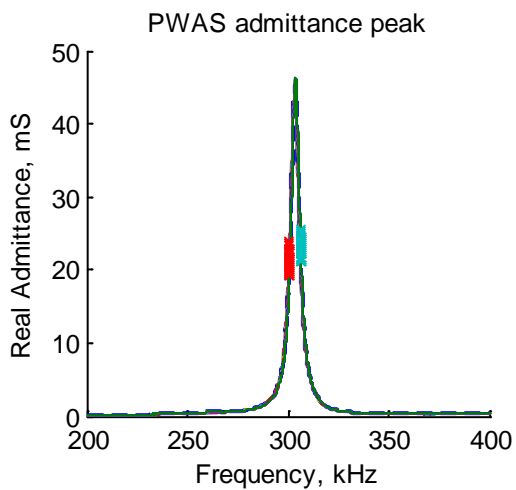
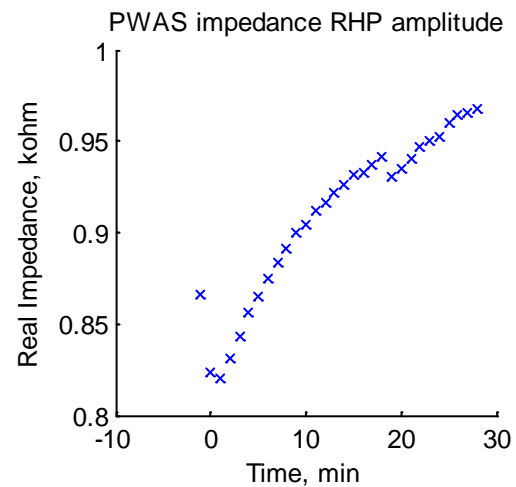
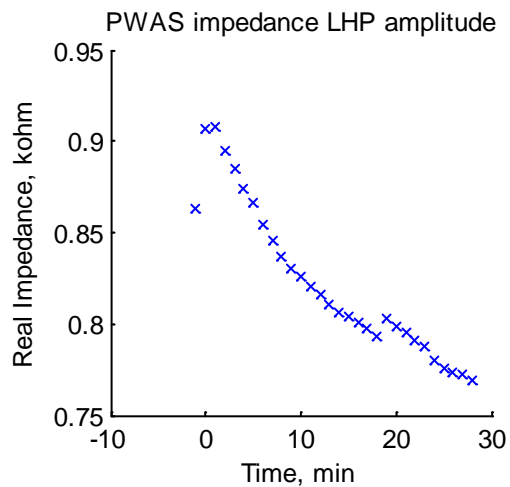
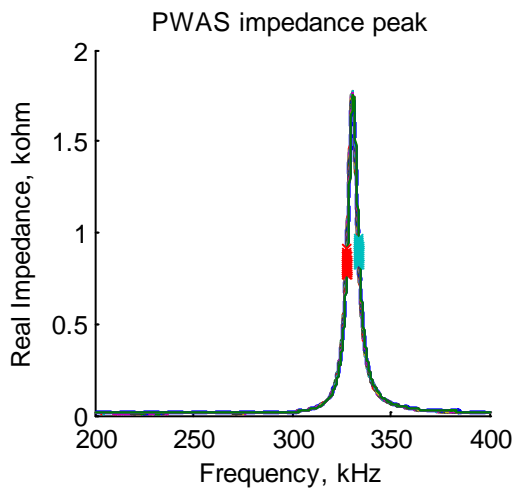


PWAS admittance peak amplitude vs. temperature PWAS admittance peak frequency vs. temperature



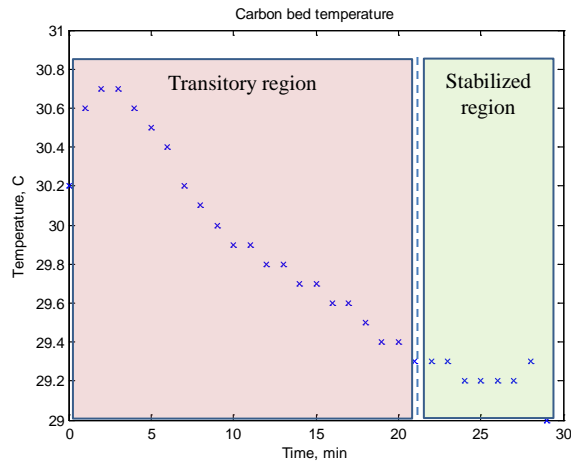
**Experiment set 1, May 29 – June 3**

*Appendix 3: 0.5% SO<sub>2</sub>, May 29*

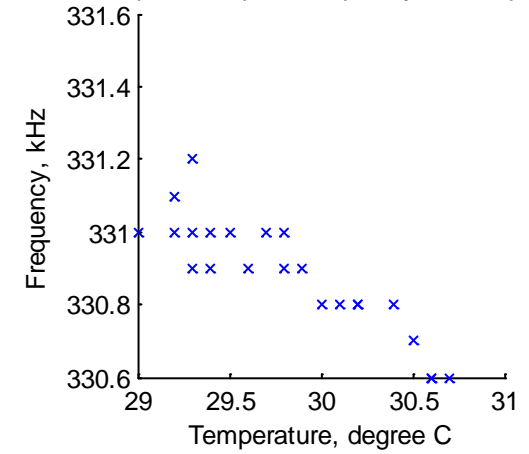
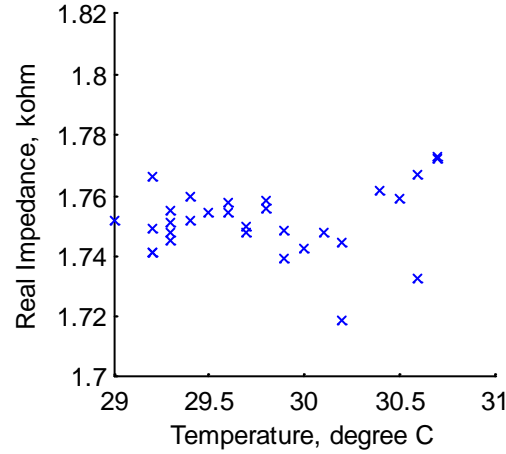


**Experiment set 1, May 29 – June 3**

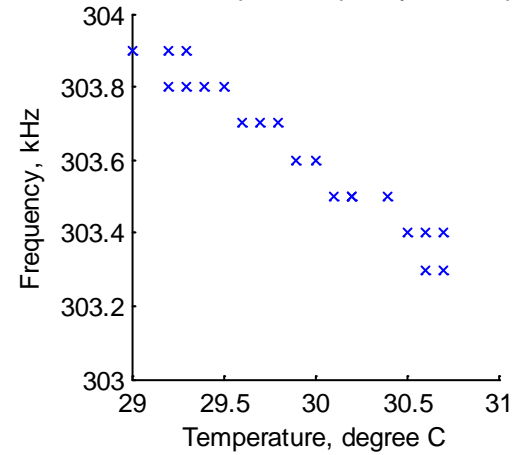
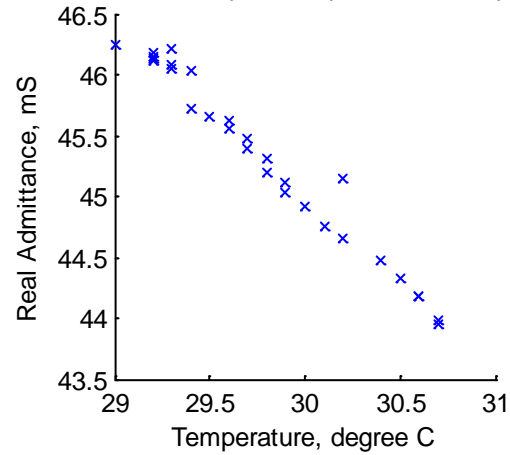
Appendix 3: 0.5% SO<sub>2</sub>, May 29 (cont.)



PWAS Impedance peak amplitude vs. temperat PWAS Impedance peak frequency vs. tempera

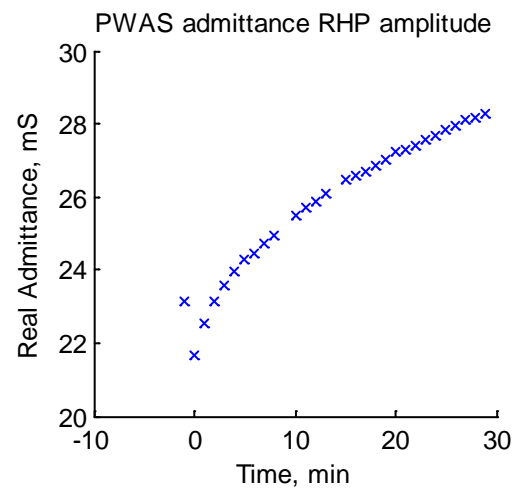
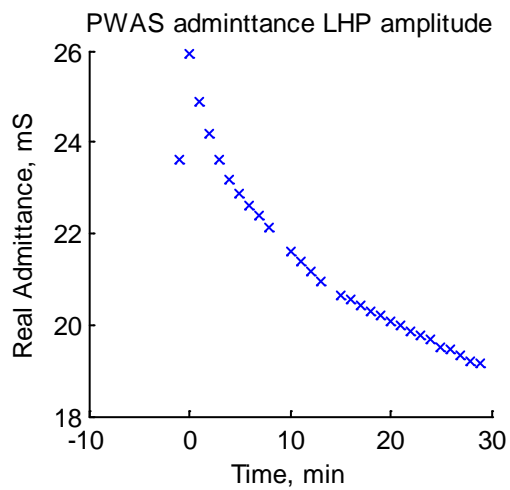
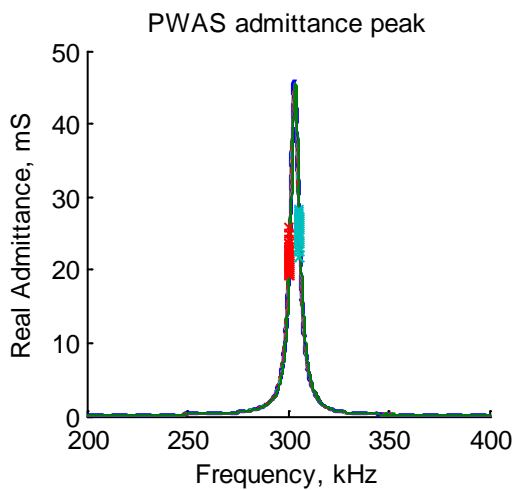
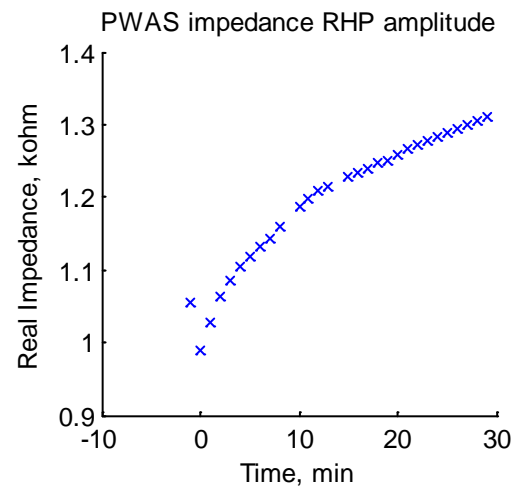
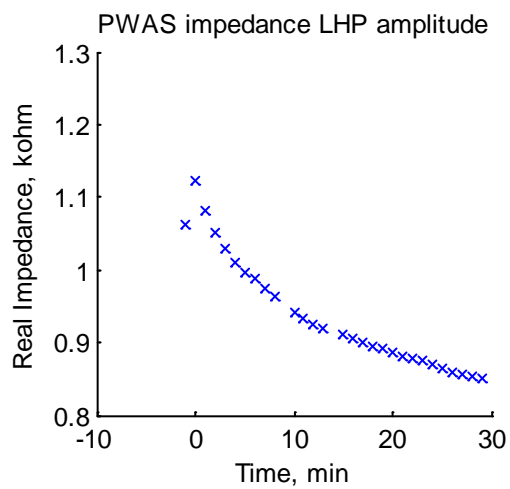
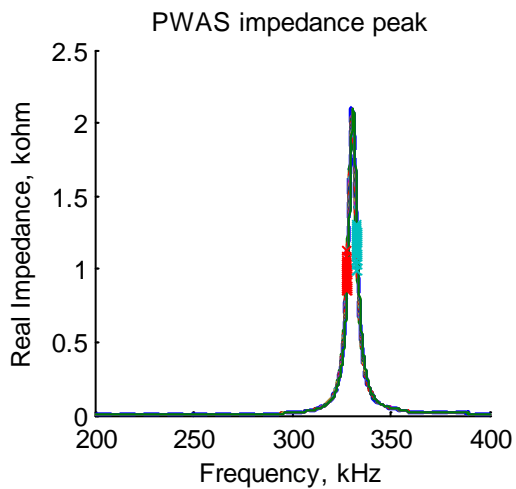


PWAS admittance peak amplitude vs. temperat PWAS admittance peak frequency vs. tempera



**Experiment set 1, May 29 – June 3**

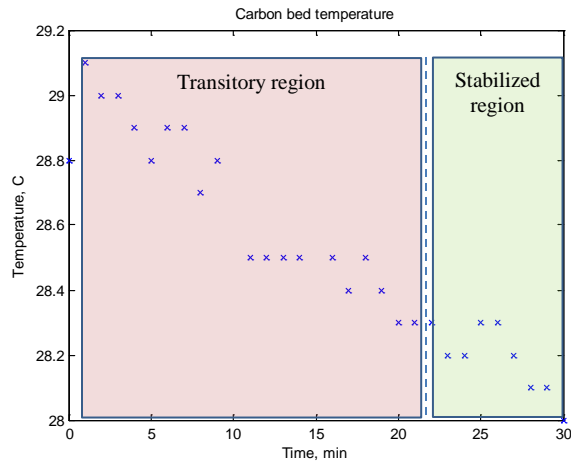
Appendix 4: 0.5% SO<sub>2</sub>, Jun 03



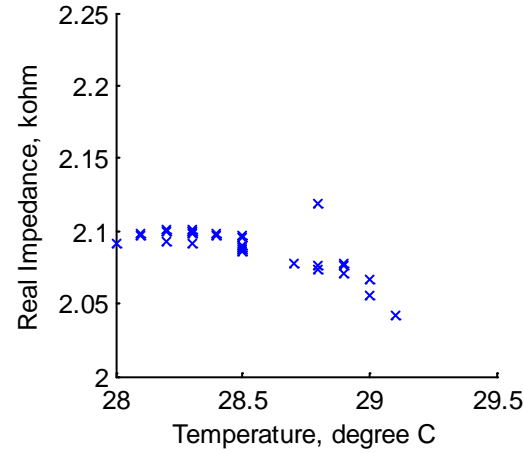


**Experiment set 1, May 29 – June 3**

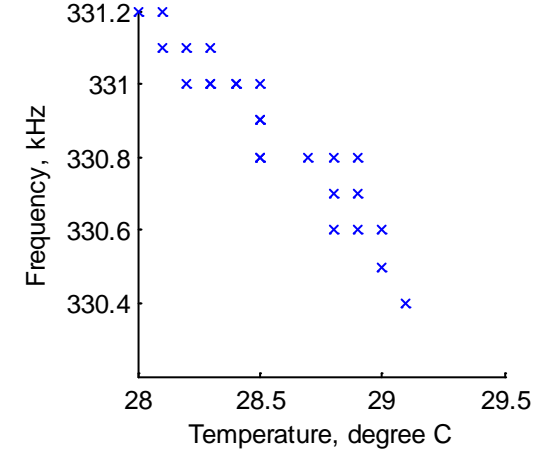
Appendix 4: 0.5% SO<sub>2</sub>, Jun 03 (cont.)



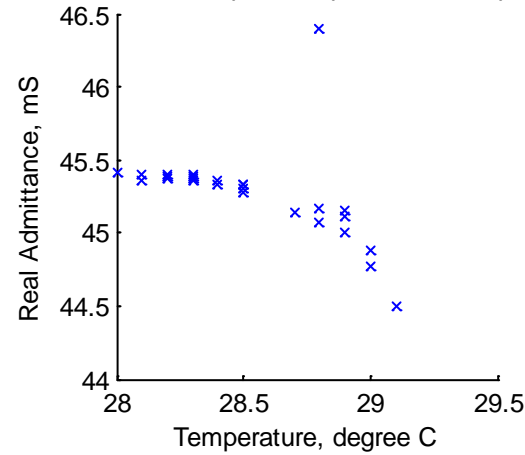
PWAS Impedance peak amplitude vs. temperature



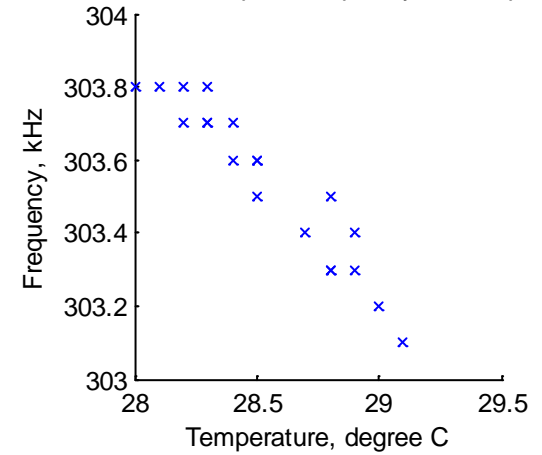
PWAS Impedance peak frequency vs. temperature



PWAS admittance peak amplitude vs. temperature

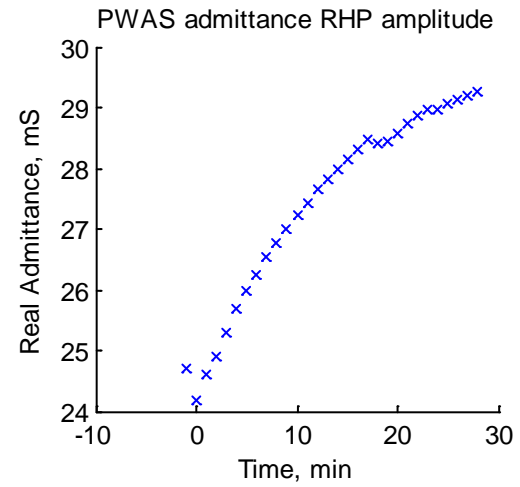
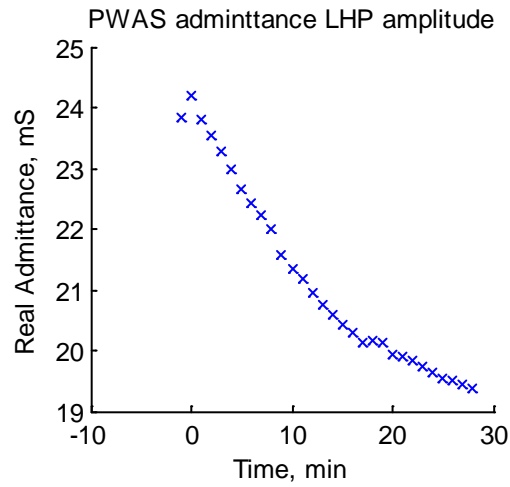
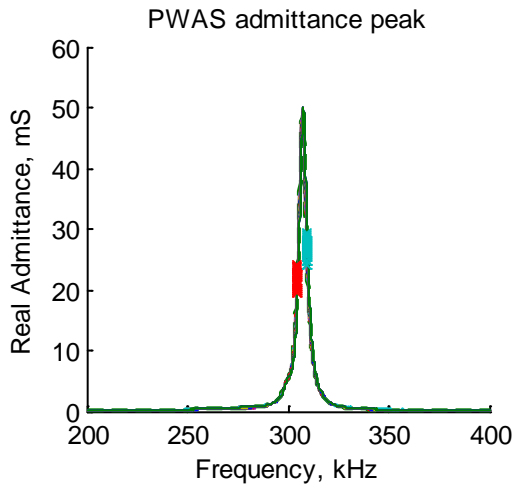
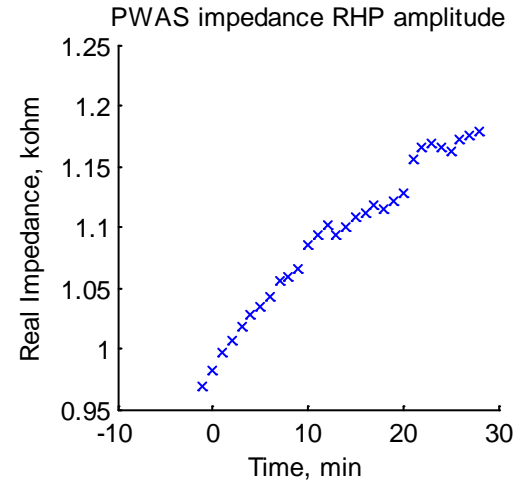
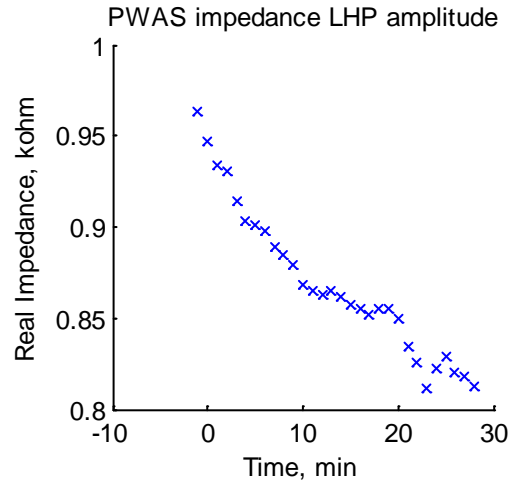
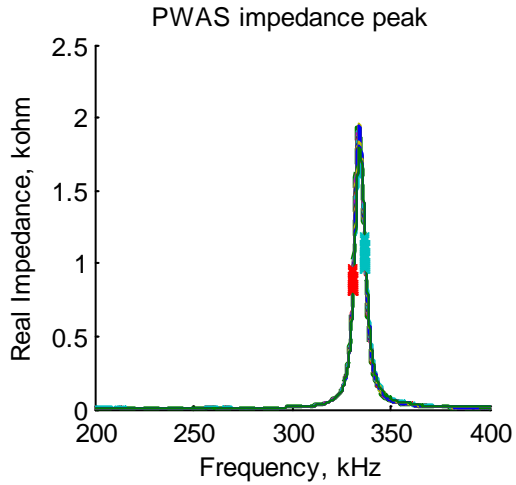


PWAS admittance peak frequency vs. temperature



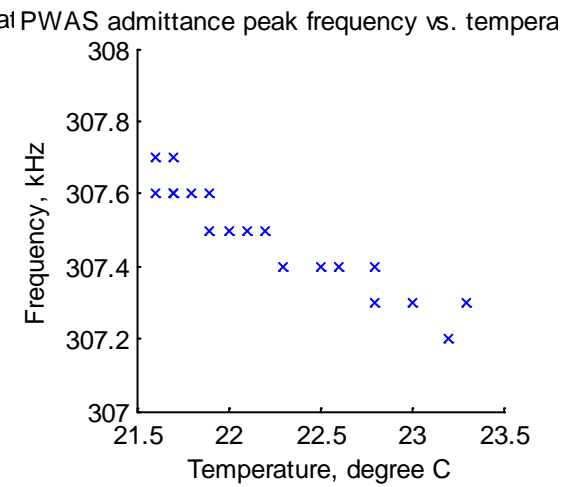
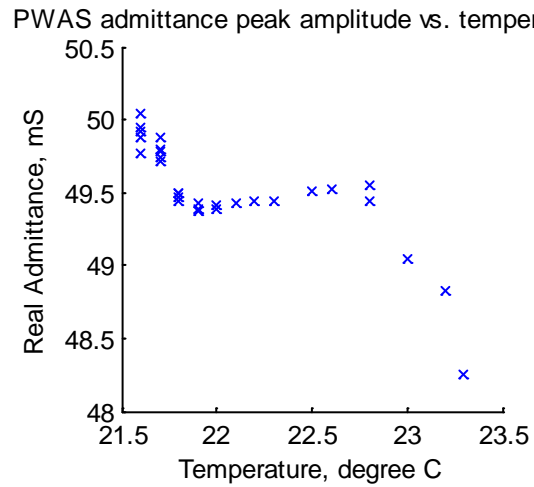
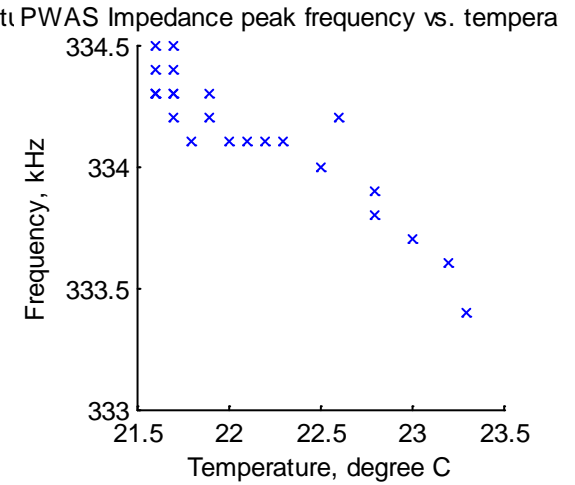
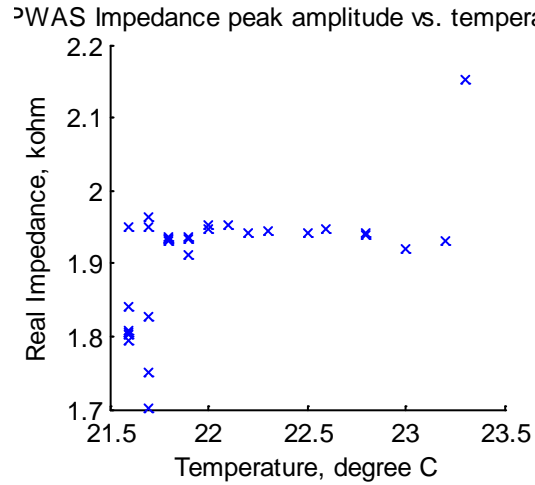
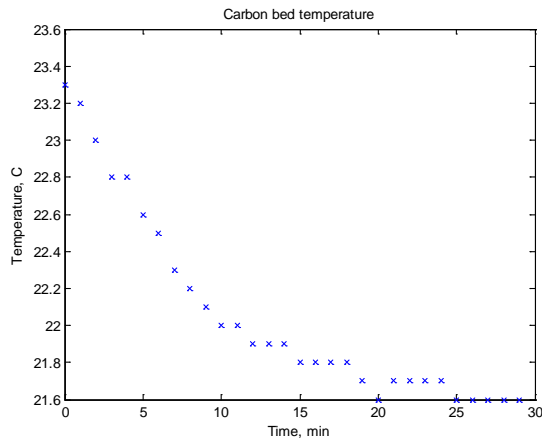
**Experiment set 1, June 18 – June 19**

*Appendix 5: Temperature test, Jun 18*



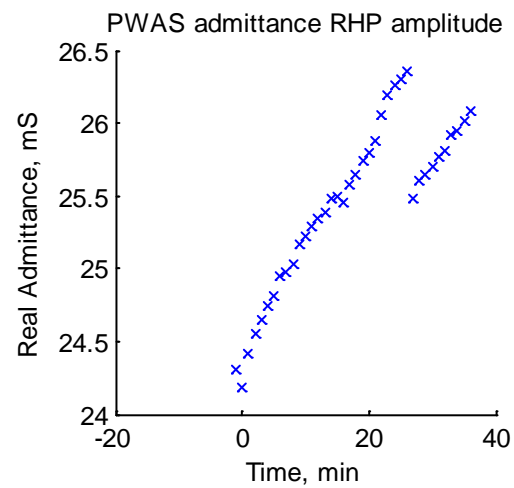
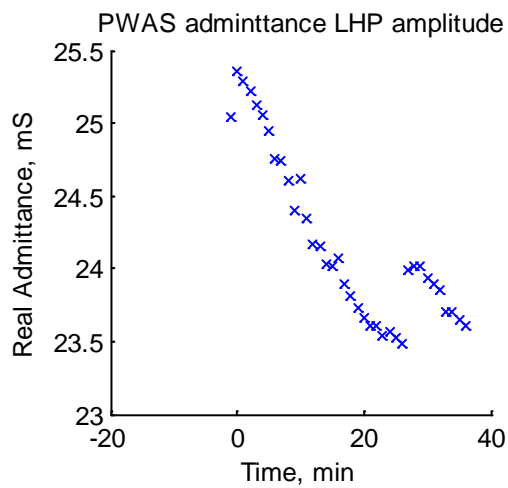
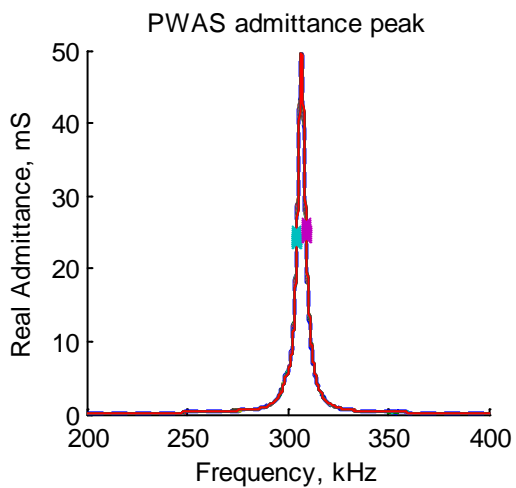
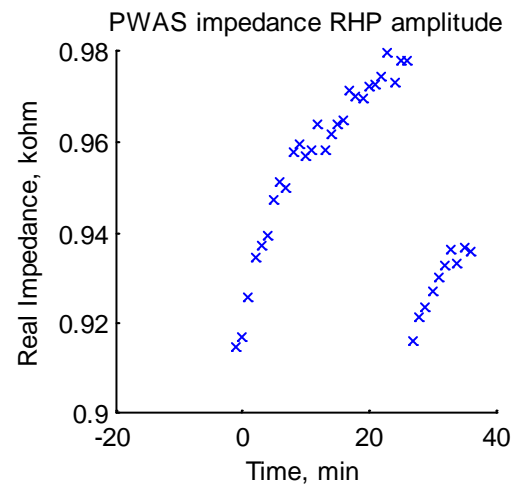
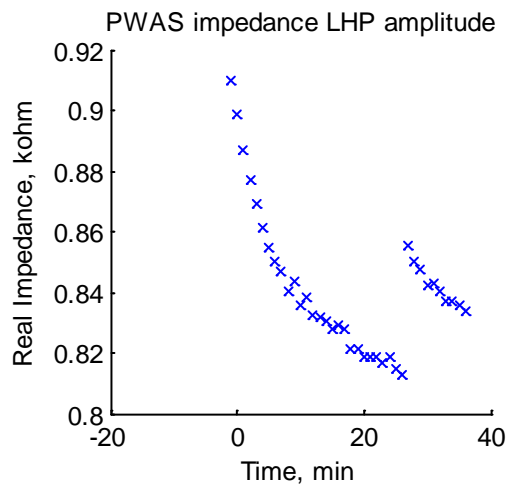
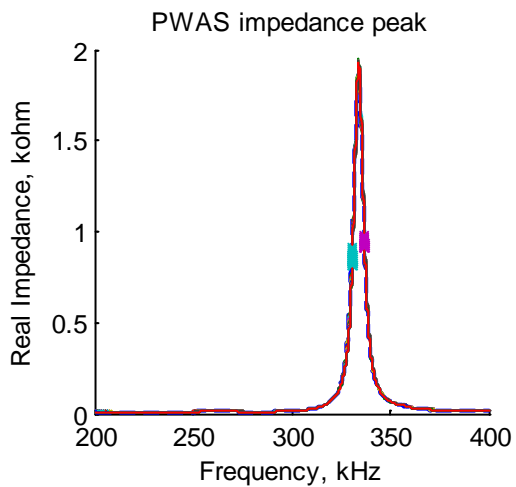
**Experiment set 1, June 18 – June 19**

*Appendix 5: Temperature test, Jun 18 (cont.)*



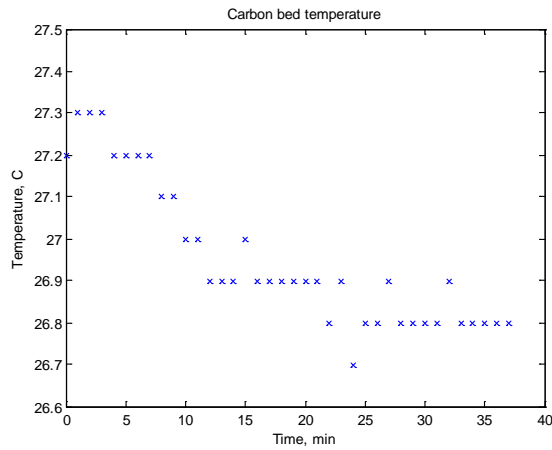
**Experiment set 1, June 18 – June 19**

*Appendix 6: SO<sub>2</sub> test, Jun18*

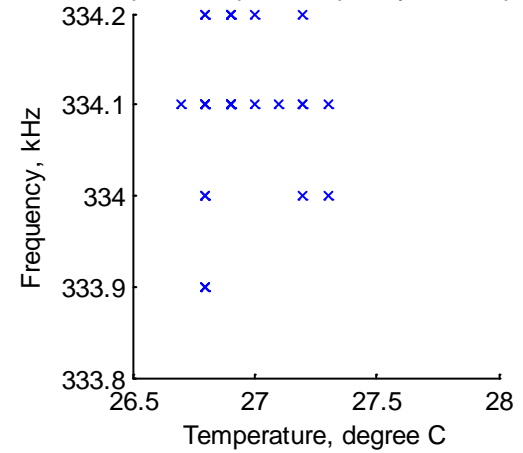
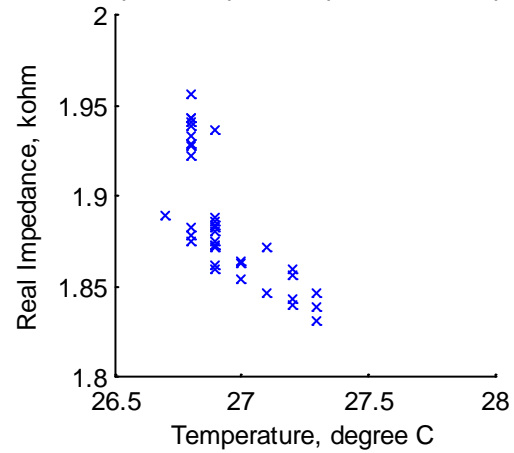


**Experiment set 1, June 18 – June 19**

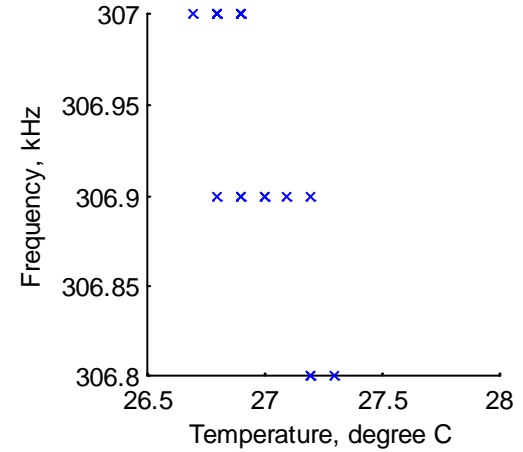
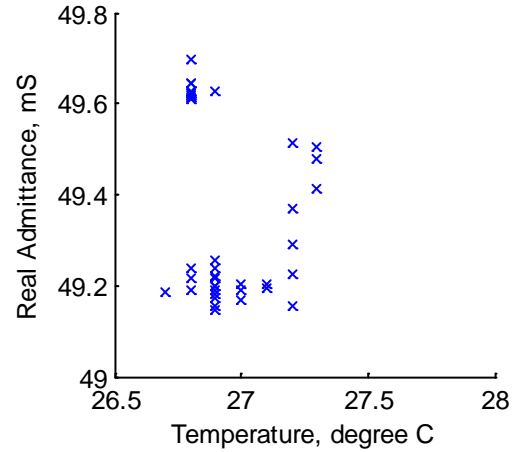
Appendix 6:  $SO_2$  test, Jun18 (cont.)



PWAS Impedance peak amplitude vs. temperat PWAS Impedance peak frequency vs. tempera

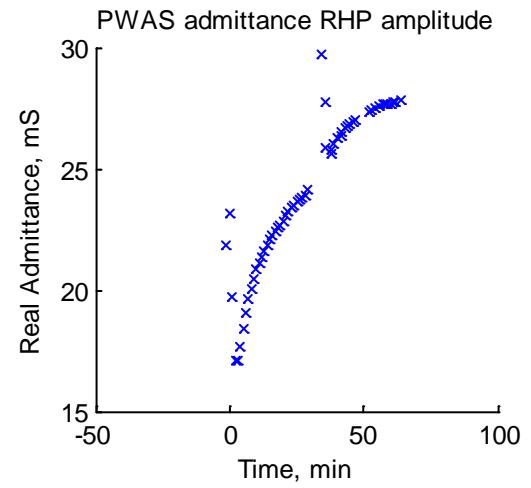
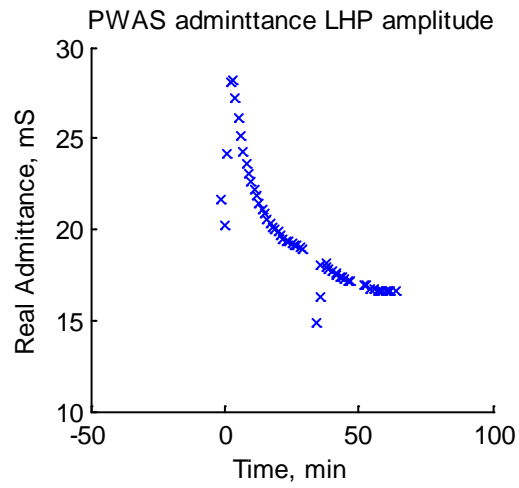
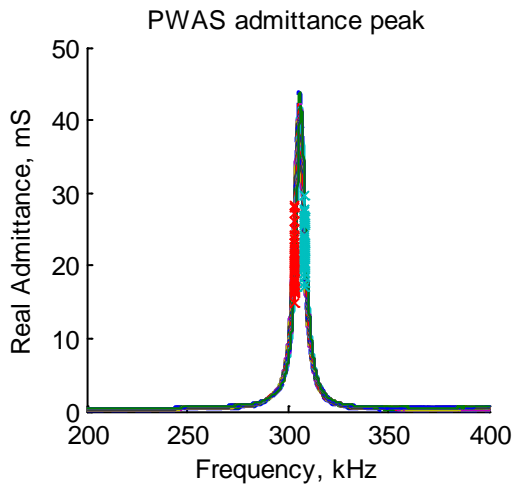
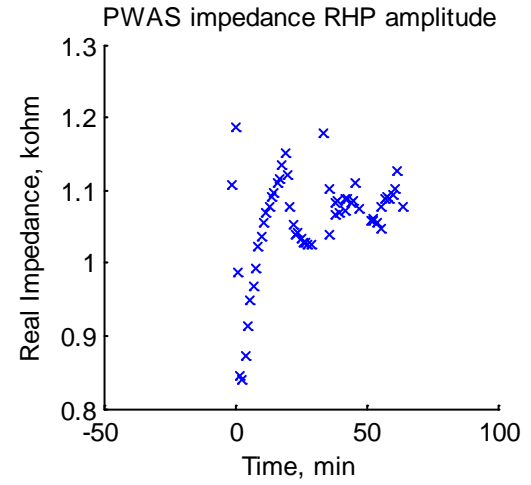
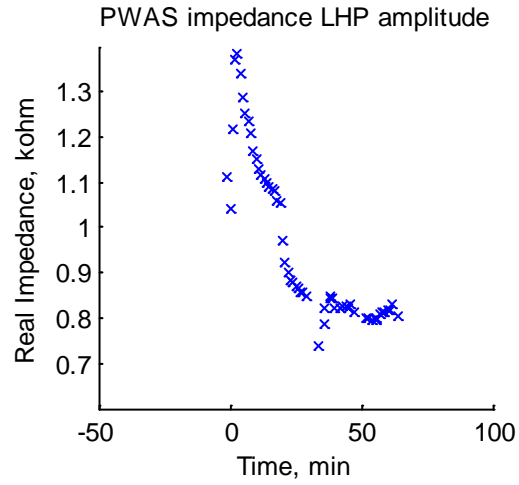
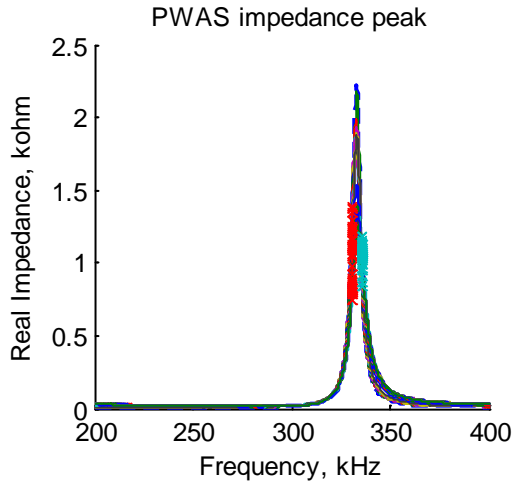


PWAS admittance peak amplitude vs. temperat PWAS admittance peak frequency vs. temper:



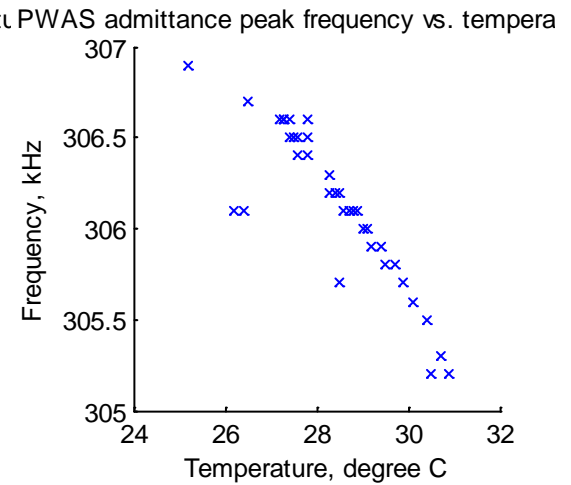
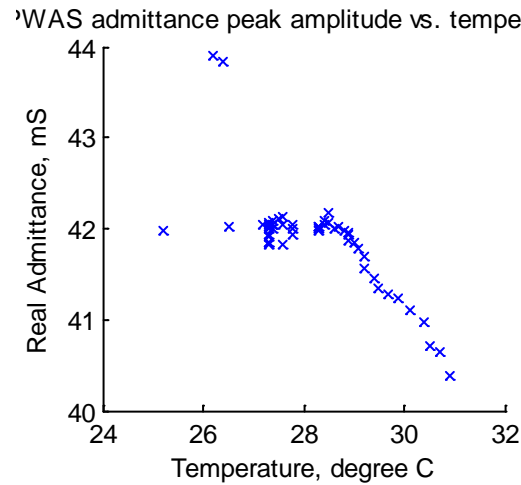
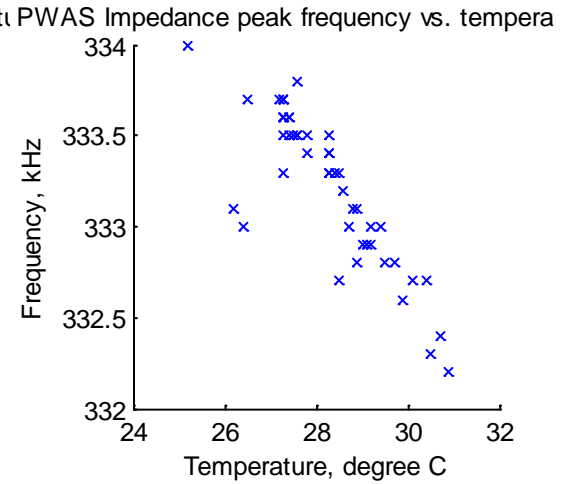
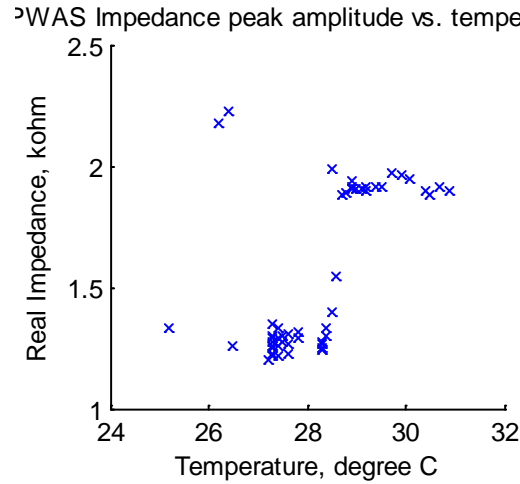
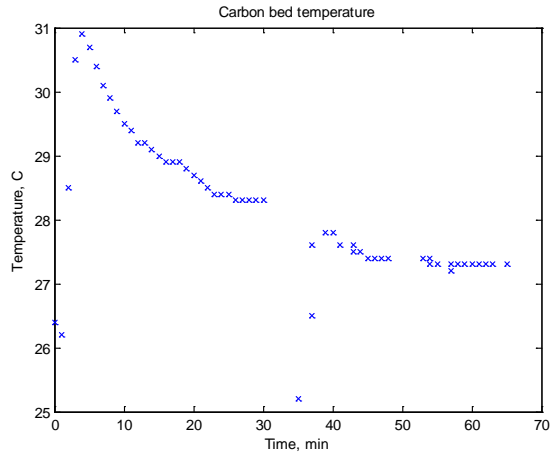
**Experiment set 1, June 18 – June 19**

Appendix 7:  $N_2$  test+ $SO_2$  test, Jun 19



**Experiment set 1, June 18 – June 19**

Appendix 7: N<sub>2</sub> test+SO<sub>2</sub> test, Jun 19 (cont.)



# **Quarterly REPORT**

**For the period January - March 2014**

## **PWAS EMIS-ECIS Active Carbon Filter Residual Life Estimation Methodology**

Submitted by:

Victor Giurgiutiu and Jingjing Bao  
University of South Carolina  
300 Main Street, Columbia SC 29208  
Telephone: (803) 777-8018  
Email: victorg@sc.edu

Submitted to:

Gregory W. Peterson  
CBR Filtration Branch  
U. S. Army Edgewood Chemical Biological Center  
5183 Blackhawk Road, Bldg. 3549  
APG, MD 21010  
Telephone: (410) 436-9794  
Email: gregory.w.peterson@us.army.mil

Date March 28, 2014

Project Number: W911NF\_11\_1

Capability Area: CB filtration



## Contents

1	Project and report overview.....	2
2	Progress during the reporting period (January-March 2013).....	3
2.1	Evaluate and improve the carbon bed SO <sub>2</sub> contamination testing apparatus.....	3
2.2	Perform carbon bed humidity test with embedded PWAS.....	5
3	Planned activities for next quarter (April-June 2014).....	9

### 1 PROJECT AND REPORT OVERVIEW

In this reporting period, we focused on two major activities: (a) evaluate and improve the carbon bed SO<sub>2</sub> contamination testing apparatus, and (b) perform carbon bed humidity test with embedded PWAS. The two activities were performed to help finalize the EMIS-ECIS bench top demonstrator design and construction. Multiple experiments were performed during the period, and data analyses were applied to identify the effects of humidity level change in the carbon bed on the PWAS electromechanical impedance spectrum.

#### Evaluate and improve the carbon bed SO<sub>2</sub> contamination testing apparatus

In the previous reporting period, we have constructed the carbon bed SO<sub>2</sub> contamination testing apparatus. Before we could start actual testing of the carbon bed with SO<sub>2</sub> contaminant, we found the SO<sub>2</sub> regulator is malfunction. This unexpected event led us to the review of our design. Through careful investigation, we found water or water vapor back flow, combined with the SO<sub>2</sub> in the mixture likely to be the cause of the damage to the regulator. Several improvements were implemented to prevent such event from happening again, such as introducer additional cut-off valves between the mixing tube and the regulators, improve the testing operation procedures.

We rebuilt the apparatus after these improvements were made and started the experiments on the carbon bed by introducing humidified N<sub>2</sub> as the first step.

#### Perform carbon bed humidity test with embedded PWAS

With the improved apparatus, we started carbon bed humidity test with embedded PWAS. This is an important step towards the final goal of this project. We intended to find out the effect of humidity level in the carbon bed on the PWAS electromechanical impedance spectrum. Previous experiments carried out in the first phase of this project have demonstrated that the introduction of water into the carbon bed will affect the EMIS by changing its resonance peak frequency and amplitude. At this stage of the project, better controlled condition, more realistic experimental parameters, and data analysis are needed to link the humidity change to the EMIS change patterns.

In this set of experiments, we started with testing the saturator for introducing water vapor to the N<sub>2</sub> gas. Multiple N<sub>2</sub> flow rate were tested to achieve various humidity levels in the N<sub>2</sub>. Then we move forward to include the carbon bed (with embedded PWAS) to the apparatus, and performed a 30 minutes test to verify the PWAS EMIS spectrum change due to the increasing humidity level in the carbon bed.

By the analysis of the experimental data, we have the following observations:

1. Humidity level up to 90% was achieved using the saturator
2. Humidity level can be changed by altering the water level
3. Reasonable consistence of humidity level (<3%) was achieved during repeated experiments
4. PWAS impedance peak amplitude change can be associated with time the carbon bed exposed to the humidified N<sub>2</sub>
5. A saturation of carbon bed seem appears after 20 minutes humidified N<sub>2</sub> flow, and the PWAS impedance can also identify such trend

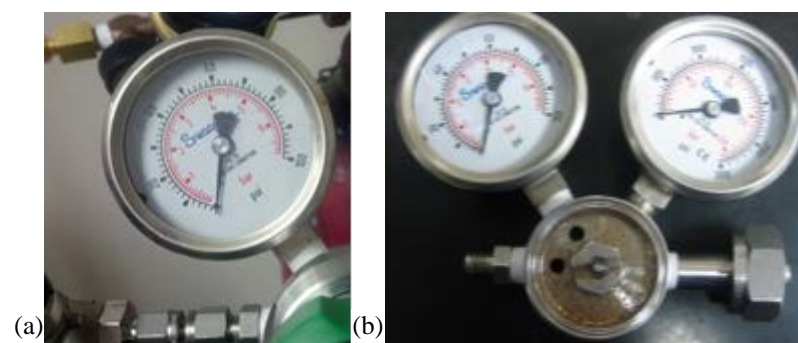
The progress details are covered in the following sections.

## 2 PROGRESS DURING THE REPORTING PERIOD (JANUARY-MARCH 2013)

### 2.1 Evaluate and improve the carbon bed SO<sub>2</sub> contamination testing apparatus

#### Investigate a malfunctioned pressure gauge on the SO<sub>2</sub> regulator, and adjust the test setup

In the beginning of this reporting period, we started testing the 0.5% SO<sub>2</sub> contamination of carbon bed. Before we could carry out the experiment, we found the pressure gauge on the SO<sub>2</sub> regulator is malfunction, and led us to the investigation of the cause of the problem. A picture of the faulty pressure gauge is shown in Figure 1 (a). The needle goes beyond the maximum range of the gauge and do not returning to zero after pressure is released. We requested the pressure gauge supplier to investigate the cause of the damage. After discussion, we suspect the regulator is not working properly, and cause the high pressure gas pass through, and cause the damage to the pressure gauge. We decide to disassemble the regulator to verify the theory. As shown in Figure 1 (b), the sealing surface of the regulator is corroded, and covered with rust. According to the analysis of the supplier, once the sealing surface is corroded, the high pressure will creep through the regulator, and directly apply to the low pressure gauge. The excessive pressure is much higher than the maximum pressure rating of the low pressure gauge, and completely damaged the gauge.



**Figure 1 Malfunctioned SO<sub>2</sub> pressure gauge and regulator: (a) the needle goes beyond the maximum range of the gauge and do not returning to zero after pressure is released; (b) the regulator was corroded.**

The regulator was made from stainless steel, and is rated as compatible with SO<sub>2</sub>. To find out the cause of the corrosion, we continued the discussion with the supplier, and reviewed our previous experiments. An observation was that the pressure level of the regulated SO<sub>2</sub> was smaller than the N<sub>2</sub> channel. There are a few occasions we found water accumulated in the SO<sub>2</sub> flow meter. It is possible that the water back flowed due to the high pressure level caused by the carbon bed. As shown in Figure 2.

We discussed this issue with a group in the chemical engineering (CHE) department in USC, and found they had similar experience when dealing with high concentration SO<sub>2</sub>. Through discussion with the group, we found a preventive measure is to add an additional cut-off valve in the SO<sub>2</sub> channel to stop possible water/vapor back flow into the flow meter and regulators.

#### Adjust the test setup and design experimental procedures

An operating procedure was designed to establish a guideline for carrying out the experiments. The following protocol defines the verification process for controlling humidity.

Getting started: Opening the nitrogen tank to the saturator

1. Open the knob on the nitrogen tank completely; this will cause the first pressure gage to read the level of pressure in the tank itself.

**Note:** if there is not reading on the first gage pressure, than the regulator has been damaged. The second gage, input pressure gage, was previous set to 30 psi. You can adjust the pressure by decreasing or increasing the pressure with the valve. However, one must be careful to have all other valve in sync completely shut closed.

2. Following opening of the nitrogen tank, open the shut-off valve that is adjacent to that of the regulator, allowing the pressure to follow the stream to the flow meter attached.

3. Open the shut-off can is adjacent to the flow meter.

**Note:** do not open the flow meter valve before the shut-off valve that leads to the saturator. This will cause the

pressure to quickly run to the saturator without accurately defining the amount of pressure that the saturator will receive.

4. Open the flow meter slightly to verify that the system is working.

Three experiments were designed to verify the capability of controlling the humidity level by the saturator.

Experiment 1: Varying flow rate.

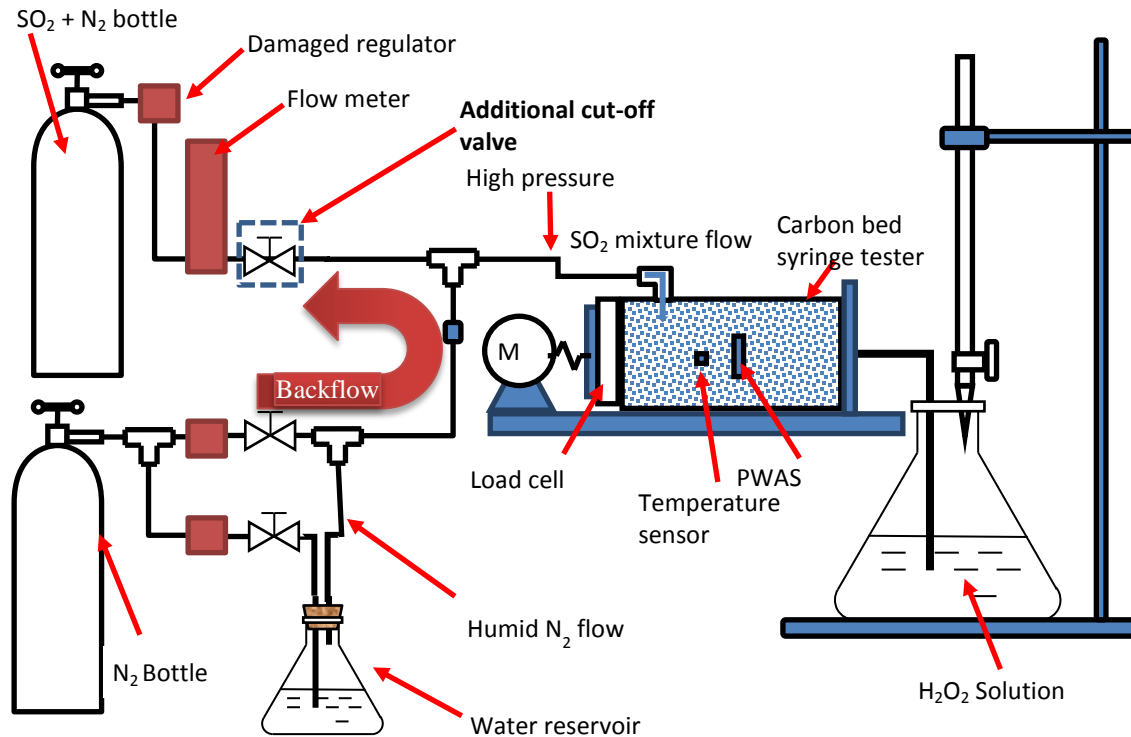
1. Fill the saturator with 100 mL of water and then replacing the frit fixture into the water.
2. Following the “Getting started” protocol, release the nitrogen gas at different flow rates and measure the relative humidity (RH%). , Turn off the flow meter after an 1 min of flow
3. Allow the humidity meter to return to the original room humidity level.
4. This procedure is repeated for the additional flow rates of various flow rates.

Experiment 2: Varying water level.

1. Similar to that of experiment 1, “Varying Flow Rate”, however, the water level was varied.
2. This was done by simply increase the water level inside the saturator. The water levels measured at 75, 100, 125, 150, 200, 225, 250, and 300 mL at a steady flow rate.

Experiment 3: Varying time.

1. Following experiment 2, RH% was measured with the variation of time
2. The water level was maximized at 200 mL with a steady flow rate. Run the test for a continuous 10 minutes, and measure the humidity level at each min interval.



**Figure 2: In current carbon bed contamination test board design, high pressure in the carbon bed may cause back flow to the flow meter and regulator. Additional cut-off valve is needed to prevent the back flow.**

## 2.2 Perform carbon bed humidity test with embedded PWAS

With the improved apparatus, we started carbon bed humidity test with embedded PWAS. This is an important step towards the final goal of this project. We intended to find out the effect of humidity level in the carbon bed on the PWAS electromechanical impedance spectrum. Previous experiments carried out in the first phase of this project have demonstrated that the introduction of water into the carbon bed will affect the EMIS by changing its resonance peak frequency and amplitude. At this stage of the project, better controlled condition, more realistic experimental parameters, and data analysis are needed to link the humidity change to the EMIS change patterns.

In this set of experiments, we started with testing the saturator for introducing water vapor to the N<sub>2</sub> gas. Multiple N<sub>2</sub> flow rate were tested to achieve various humidity levels in the N<sub>2</sub>. Then we move forward to include the carbon bed (with embedded PWAS) to the apparatus, and performed a 30 minutes test to verify the PWAS EMIS spectrum change due to the increasing humidity level in the carbon bed.

### Evaluation of introducing humidity with saturators

In the reporting period, we installed a small saturator to start testing the humidity level with the three experimental conditions explained in the previous section. In these experiments, we used a small saturator, as shown in Figure 3.

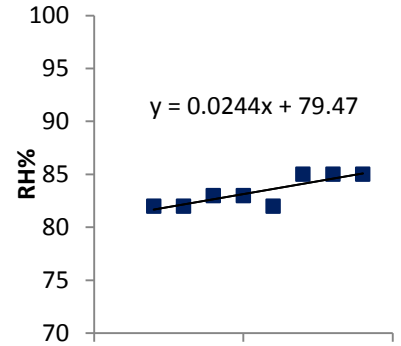


**Figure 3 Humidify the N<sub>2</sub> stream by a saturator.**

We started by changing the flow rate of N<sub>2</sub> from 90 mL/min up to 230 mL/min. The experimental data is shown below.

**Table 1 Humidity level at different flow rate**

Test	Temp. (°C)	Pressure (psi)	Time (min)	Water volume (mL)	Flow rate (mL/min)	Humidity (RH%)
1	21	30	1	100	90	82
2	21	30	1	100	110	82
3	21	30	1	100	130	83
4	21	30	1	100	150	83
5	21	30	1	100	170	82
6	21	30	1	100	190	85



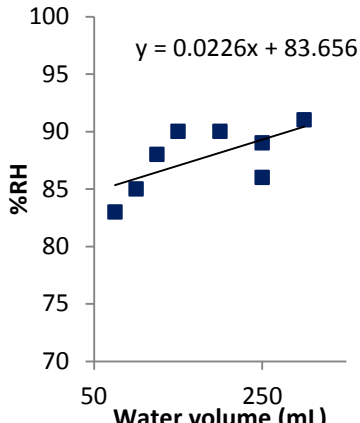
7	21	30	1	100	210	85
8	21	30	1	100	230	85

The humidity data were captured after running the humidified N<sub>2</sub> for 1 minute. At 21 °C and 30 psi input N<sub>2</sub> pressure level, the humidity level of the output N<sub>2</sub> was maintain at 82 - 85 RH% level, which is not significantly changed by the flow rate settings. It is apparent that this is the humidification level can be achieved with the saturator design.

We continue with the experiment 2, and change the total amount of water in the saturator, which will affect the distance gas travels in the water. The data is shown in the table below.

**Table 2 Humidity level at different water volume**

Test #	Temp. (°C)	Pressure (psi)	Time (min)	Water volume (mL)	Flow rate (mL/min)	Humidity (RH%)
1	21	30	1	75	170	83
2	21	30	1	100	170	85
3	21	30	1	125	170	88
4	21	30	1	150	170	90
5	21	30	1	200	170	90
6	21	30	1	250	170	89
7	21	30	1	250	170	86
8	21	30	1	300	170	91

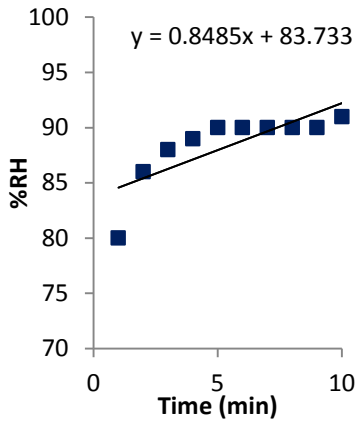


The humidity level changed from 83 RH% to 91 RH% when the water level changed from 75mL to 300 mL. It seems the long the distance the gas travels in the water, the higher the humidity level change be obtained.

Then, we performed experiment 3, and run the test for 8 minutes, and check the humidity level at each minute interval. The experimental data is shown in the table below.

**Table 3 Humidity level at different running time**

Test #	Temp. (°C)	Pressure (psi)	Time (min)	Water volume (mL)	Flow rate (mL/min)	Humidity (RH%)
1	21	30	1	200	170	80
2	21	30	2	200	170	86
3	21	30	3	200	170	88
4	21	30	4	200	170	89
5	21	30	5	200	170	90
6	21	30	6	200	170	90
7	21	30	7	200	170	90
8	21	30	8	200	170	90



The humidity level shows a graduate increase during the test. This suggests a running time around 5 minutes may be required to achieve stabilized humidity level at the output port, which may also involve the humidity reading device measurement requirement.

It is worth noting that the humidity level can change a small percent in repeated tests. For example, at 100 mL water level, 170 mL/min flow rate, and measured at 1 minute run time, two humidity data was 82 %RH (in Table 1) and

85 %RH (in Table 2), which comes to a 3.6% error. This can be caused by the resolution of the flow meter and the fluctuation of the humidity level by the nature of the simple setup.

In summary, the N<sub>2</sub> humidification setup was tested with a group of experimental setups. Multiple testing parameters were changed separately to find their relation with the humidity level at the output. After the experiments, we have the following observations:

1. Humidity level up to 90% was achieved using the saturator
2. Humidity level can be changed by altering the water level
3. Reasonable consistence of humidity level (<3%) was achieved during repeated experiments

#### **Perform humidity test on carbon bed embedded with PWAS**

After verification of the performance of the saturator, we include the carbon bed to the apparatus to test the effects of humidity level in the carbon bed on the PWAS EMIS spectrum.



**Figure 4 Experimental setup used for the humidity test on carbon bed embedded with PWAS.**

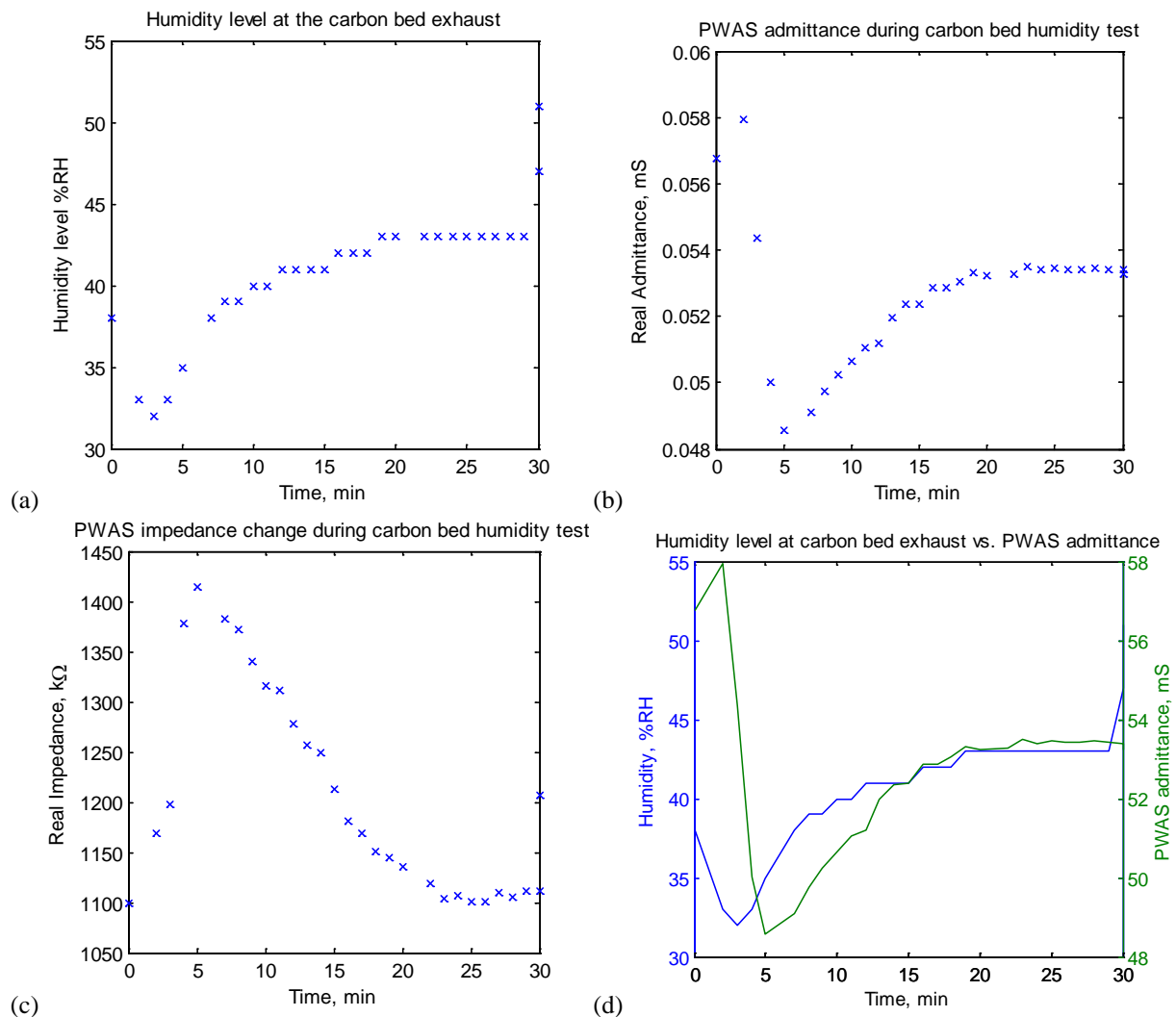
To investigate the effect of humidity on the absorption of the carbon granules, a protocol was developed to examine a simple humidity system connected to the carbon chamber. A ¼" Teflon tube was connected to a flow meter which leads to a ball valve used to regulate the gas flow. This was then joined with a 500 mL saturator with 200 mL of water where the outtake valve continued to the carbon chamber.

To begin the testing, the protocol to open the gas line was followed. Once the gas line was opened, allowing the gas to escape through the syringe, the initial humidity was taken. This is done without the carbon granules. Additionally, the environmental humidity was also recorded.

Following this, the gas line was closed with the ball valve and the carbon granules were placed inside the chamber, and the plunger with the PWAS was inserted. The impedance was recorded. The gas line reopened following the steps as stated previously. At this point, impedance and humidity was recorded at each one minute interval continuously up to 30 minutes.

The following steps were taken during the experiment:

1. Open the gas line using protocol previously presented.
2. Measure the initial humidity of the surrounding environment and escaping gas in the syringe without any carbon granules.
3. Close the gas line. Fill the syringe up to 30 mL of carbon granules and insert the plunger with the PWAS into the syringe, careful on to damage the PWAS.
4. Record the initial impedance of the PWAS within the carbon granules.
5. Reopen the gas line and record impedance at one minute intervals continuously for 30 minutes.



**Figure 5** Experimental data from the humidity test on carbon bed embedded with PWAS. (a) Humidity level measured at the carbon bed exhaust, (b) the PWAS admittance peak value during the experiment, (c) the PWAS impedance peak value during the experiment, and (d) the comparison between the humidity level at carbon bed exhaust and the PWAS admittance peak value.

Total of 29 data points were taken during this experiment, and the experimental data are shown in Figure 5

It can be noticed from Figure 5(a), the humidity level detected at the carbon bed started at low level (32%RH) from the beginning of the experiment, then increases with the testing time. At around 17 minutes into the test, the humidity level seems reached and maintained at a high level of 43%RH. On the PWAS admittance plot Figure 5(b), similar trend was noticed, the admittance start from close to 0.048mS at 5 minute, and increases with the time of experiment, until it reaches about 0.053mS at about 17 minutes, and keeps almost constant till the end of the experiment. The admittance is the inverse of impedance, hence in Figure 5(c), the trend of PWAS impedance shows a reversed pattern. In the last plot Figure 5(d), the humidity level at the exhaust of the carbon bed is compared with the PWAS admittance level. and a good correlation between the two variable are noticed.

From the experimental plot, the following can be observed:

1. PWAS impedance peak amplitude change can be correlated with time the carbon bed exposed to the humidified N<sub>2</sub>
2. A saturation of carbon bed seem appears after 20 minutes humidified N<sub>2</sub> flow, and the PWAS impedance can also identify such trend

### **3 PLANNED ACTIVITIES FOR NEXT QUARTER (APRIL-JUNE 2014)**

For the upcoming quarter (January-March), we will continue work on testing the carbon bed SO<sub>2</sub> contamination apparatus (Task 4), and on: Specifications for prototype development and final report (Task 5). We will report to ECBC specialists the progress of the tasks, and discuss the brass board design draft. Once the design gets approval, we will start constructing the brass board tester.

In the next reporting period, we will move to mix SO<sub>2</sub> with the humidified N<sub>2</sub>, and perform SO<sub>2</sub> contamination tests on the carbon bed with embedded PWAS. The PWAS impedance/admittance spectrum will be collected with the humidity and SO<sub>2</sub> level. The various contamination scenarios will be performed. Data analysis will be performed on the experimental data. The results will be used to assist the finalization of the EMIS-ECIS bench top demonstrator design and construction

#### **Perform Task 4: EMIS-ECIS bench top demonstrator design and construction**

This task will focus on designing and constructing an integrated bench top experimental setup that will demonstrate the different aspects of the EMIS-ECIS methodology. We will use the design of specialized experiments from Task 2, extend them, and then assemble them into a multifunction platform. The design will be adapted to the model development. A breadboard system will be developed for the demonstrator.

#### **Perform Task 5: Specifications for prototype development and final report**

In this task, we will develop specifications for a prototype to transition the research results and fully implement them into an actual piece of equipment. As we mentioned earlier, the focus of the present project is to understand the PWAS-carbon bed impedance model; full development of residual life estimation system will need further efforts. The methodology developed in this project should be transitioned to practice and implemented into a proof of concept prototype to address the issues Army CBR Filtration concern. We will provide the specifications of such a prototype, to fully demonstrate the capacity of proposed methodology, and pave the way to the next step of development. In this task, we will also compile the comprehensive final report of the project.