

The Pennsylvania State University

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Final Report for Phase III

Neutron Computed Tomography of Freeze/thaw Phenomena in Polymer Electrolyte Fuel Cells

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1. Summary

This report summarizes the final year's progress of the three-year NEER program. The overall objectives of this program were to 1) design and construct a sophisticated high-resolution neutron computed tomography (NCT) facility, 2) develop novel and sophisticated liquid water and ice quantification analysis software for computed tomography, and 3) apply the advanced software and NCT capability to study liquid and ice distribution in polymer electrolyte fuel cells (PEFCs) under cold-start conditions. These objectives have been accomplished by the research team, enabling a new capability for advanced 3D image quantification with neutron imaging for fuel cell and other applications. The NCT water quantification methodology and software will greatly add to the capabilities of the neutron imaging community, and the quantified liquid water and ice distribution provided by its application to PEFCs will enhance understanding and guide design in the fuel cell community.

The previous two years' program accomplishments were summarized in the reports for the corresponding years. In this report, a focus on the final year accomplishments is given with a brief summary of the Year 1 and Year 2 accomplishments. During the third year of the project, significant progress has been made to enable the accomplishment of the program's goals. Specifically, during the past year:

- 1) A newer ^6Li based scintillation screen was installed in the imaging system. This scintillation screen provides better image quality and resolution over the GdO_2S screen,
- 2) A water quantification technique for NCT imaging was developed and tested, and
- 3) Aluminum test samples were cooled to sub-zero temperatures and then filled with known water amounts, which froze, to mimic ice formation in fuel cell flow fields.

The amount of ice present was successfully quantified with the NCT water quantification technique, enabling regular testing of frozen fuel cells to be accomplished.

This progress comes on top of the first and second year results, where the first set of images of a frozen fuel cell were obtained, specialized tomography software was acquired for image reconstruction, initial experiments with specially designed dummy cells were successfully performed with full image reconstruction, and the implementation of completely new neutron imaging and fuel cell facilities.

2. Task Summaries

The original objectives of this research were to:

- 1) Design and construct a sophisticated high-resolution neutron computed tomography (NCT) facility,
- 2) Develop and test sophisticated liquid water and ice distribution observation and quantification method and analysis software for CT (applicable for all computed tomography, applied here for neutron CT), and
- 3) Apply the advanced software and NCT capability to quantify the ice distribution in polymer electrolyte fuel cells (PEFCs) under cold-start conditions.

The overall progress towards these objectives has been substantial and successful, as detailed in the following section. All technical objectives have been met, although visualization in operating fuel cells is not yet completed. Instead, focus has been placed on demonstrating and improving quantification techniques on simplified dummy fuel cell designs, so that the approach can be validated, and utilized on fuel cells and other systems when the research need arises. The system is now fully capable of visualization with accurate quantification in operating fuel cells, as described in this report.

2.1 Development of Computed Tomography and Fuel Cell Facilities - Objective #1 Completion

2.1.1: Fuel Cell Facilities

Progress along the originally scheduled Task 1 progressed on schedule during the first year of the project. During the second year of the project, additional changes to the fuel cell facilities were undertaken. Specifically, two recirculation chillers were installed to provide active cooling of the fuel cell above and below 0°C and a remote pneumatically activated test stand (Fig. 1. and Fig. 2) was added to allow 2-D imaging of larger fuel cells. During the third year of the program, additional improvements were made to integrate a National Instruments Labview data acquisition and test control system into the test system for easy of testing.

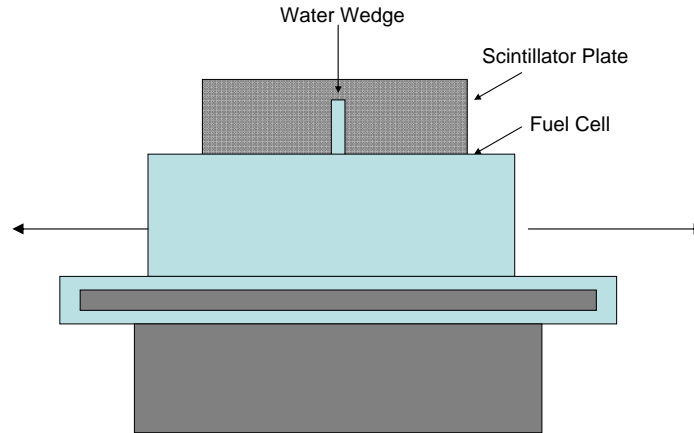


Fig. 1. Sketch of the pneumatic remote control test stand. Fuel cell moves along a pneumatic rail.

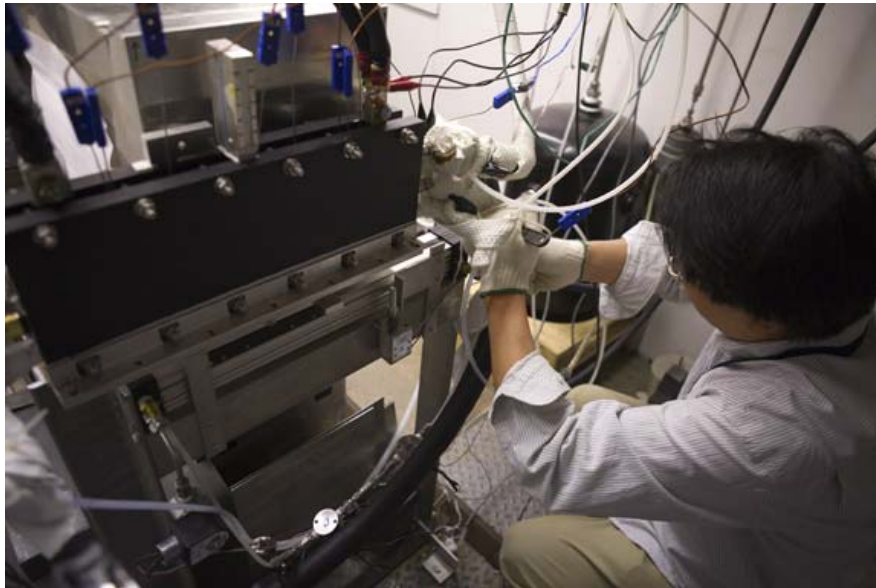


Fig. 2. Ph.D. Student works to install a fuel cell on pneumatic remote control test stand.

2.1.2 Neutron Imaging Facilities

During the first year of this project the neutron imaging facility was significantly upgraded with the addition of a Cohu 7700, 10 bit, 1004 x 1004 pixel array CCD camera and installation of a GdO_2S scintillator, and a new neutron computed tomography system, which included a new computer system for data acquisition and reduction and special tomographic reconstruction software. During the second year, enhancements to the system were required and consisted of replacing the first year CCD camera with a Retiga 4000RV 12-bit, 2048 x 2048, pixel array CCD camera, a 25.4cm x 25.4cm (10inch x 10inch) square GdO_2S scintillation screen (a 57% increase in viewing area over the year one screen) and a redesigned housing to reduce the effects of radiation on captured

images The entire housing is light tight and has been designed according to the boundary conditions given by the size and shape of the individual detector components as well as the desired image size and the space available at the facility [1]. Figure 3 illustrates the layout of the existing system.

During the third year, a new 25.4cm x 25.4cm (10inch x 10inch) ^6Li based scintillation screen was installed to replace the year two GdO_2S screen. The GdO_2S scintillation screen, while able to produce quality images for stationary objects, exhibited a “ghosting” effect for objects that were moved. After an object was removed from the viewing area, the scintillation screen produced a faint outline of the object, which remained for several minutes. Since NCT requires the revolution of an object, the scene in an image is always changing and the ghosting affect on the image would result in erroneous reconstruction results. The new ^6Li based screen rectifies the problem.

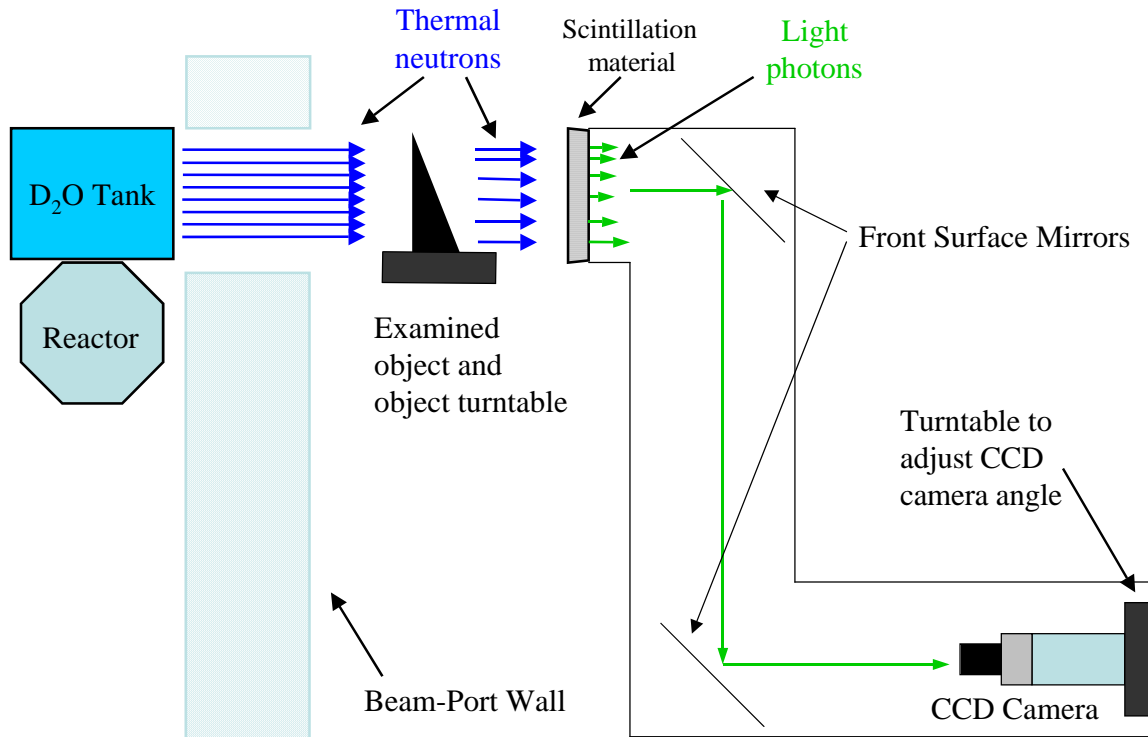


Fig. 3. Sketch of the neutron computed tomography system

2.1.3: Optimization of Neutron Computerized Tomography System

During the first year of the program, the team purchased a new data processing computer, along with the neutron tomography reconstruction software Octopus 8.0. A 3-D visualization program, VG Studio Max 1.2 was also obtained. A precise rotary table (a Newport 855C rotary table controller with resolution of 0.001°) was installed with remote computer synchronization so that a sequence of digital images from different view angles can be taken automatically and stored on the computer system. Sample data were taken of a dummy system to verify proper reconstruction was achievable. We also identified

the image resolution achievable, and determined that excessive radiation exposure of the CCD camera used limited the potential for the old optical system to achieve the desired resolution needed for 3-D imaging of the fuel cell.

During the second year, a completely new imaging system, as described above, was designed, purchased, or built, installed, and verified. A sample image taken of a fuel cell under operation is given in Fig. 4. The level of detail is much improved over the previous system (compare Figures 4 and 5), the difference in attenuation of the channels and lands due to the different material thicknesses can be easily seen (this was not possible with the old system), and white spots from radiation damage to the camera are also eliminated.

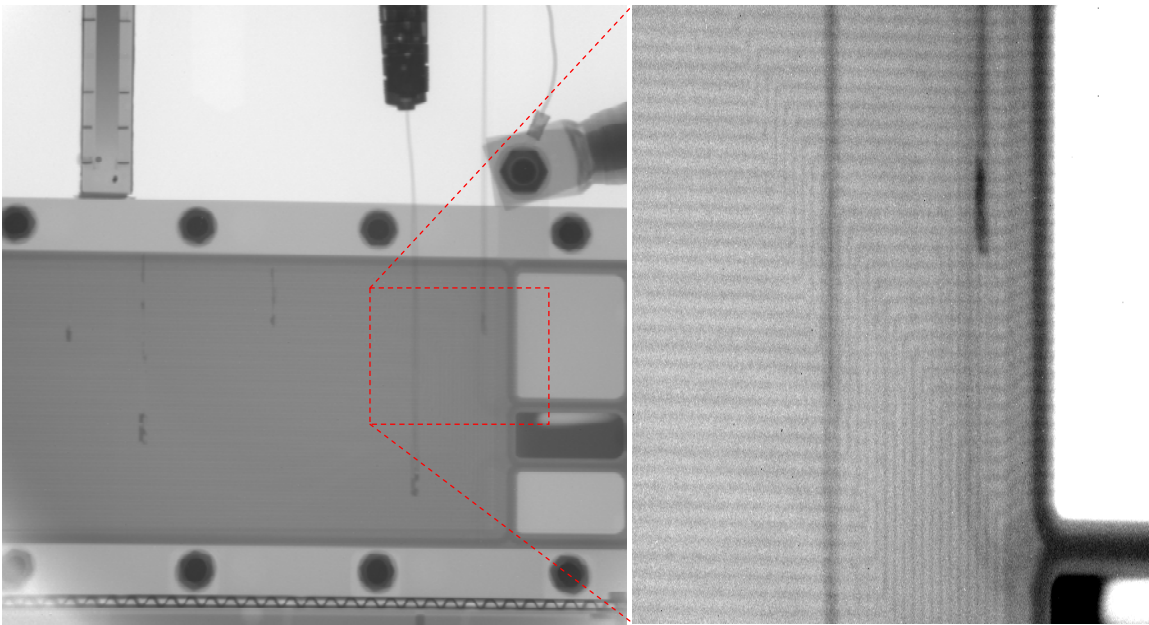


Fig. 4. 2-D radioscopic image acquired with the newly upgraded imaging system

A picture from the old system for comparison is given in Fig. 5. Note that the channels observed in this image are 400% wider than those in Fig. 4, and over 100% deeper in the through-plane direction. From this image, there are many more white spots due to radiation damage, and it is impossible to see the fine details under the large channels without false coloring.

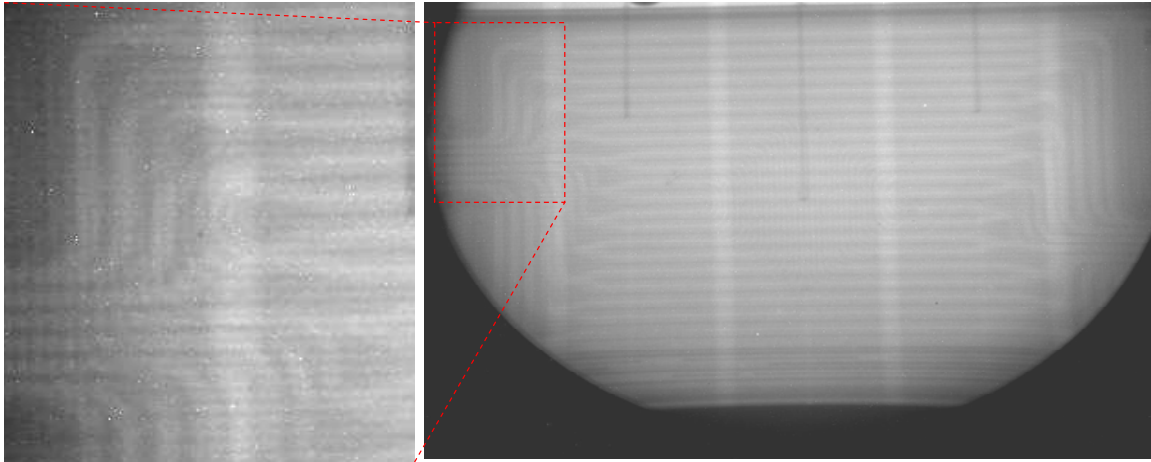


Fig. 5. 2-D radioscopic image acquired with the old imaging system

In order to conduct NCT testing with precise water quantification, the generally planar fuel cells used must be replaced with a cylindrical design to reduce geometric unsharpness. A cylindrical fuel cell with specialized membrane electrode assemblies was acquired from Prof. Michael Hickner at Penn State, who had used the cell for planar imaging at NIST. The cylindrical design is perfect for NCT. The only modification that was made was the installation of cooling channel plates on the planar walls of the fuel cell shown in Fig. 6 to enable accurate temperature control and freezing conditions. A remote coolant purge system was designed and is in place to remove the glycol mixture before each slice imaging.

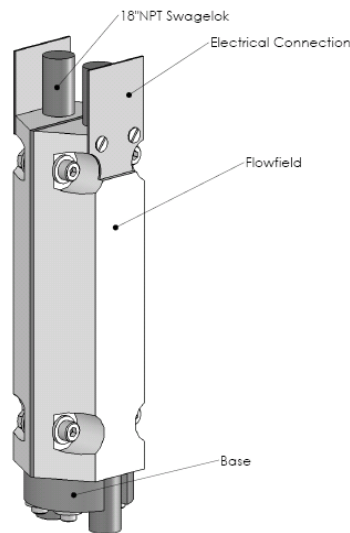


Fig. 6. CAD Drawing of NCT fuel cell acquired for testing.

2.2. Liquid Water and Ice Distribution Quantification Method - Objective #2 Completion

2.2.1 Methodology

One of the major objectives of the program was the development and testing of a water quantification technique for NCT. This has been successfully accomplished and is based on calculating the water mass represented by a voxel using a known reference: a voxel whose water mass is known. A brief synopsis of this technique follows:

A 2-D radioscopic image is comprised of individual picture elements, or pixels, whose gray levels are indicative of neutron attenuation. A 3-D volumetric reconstruction is comprised of individual volume elements, or voxels. The gray level of a voxel is representative of neutron total macroscopic cross section, Σ_t , for a given material. If the gray levels of two voxels are different it usually means they represent two different materials. However, sometimes voxels may have two different gray levels, but represent the same material. This can happen if the amount of mass of the material changes from one voxel to the next. This can be thought of as a voxel that is “partially filled” by a material. This typically occurs at material boundaries. A water quantification technique must calculate the amount of water mass in each voxel, including the partially filled ones, to be accurate.

First, the effects of all materials present other than water must be removed. This can be accomplished through background normalizing each projection image in the same manner as is often done in 2-dimensional radioscopic water quantification [2, 3]. The resulting 3-D reconstruction will yield structures comprised of water only. This also has the affect of ensuring any change in gray level from one voxel to another is the result of a change in water mass because all other materials have been effectively eliminated.

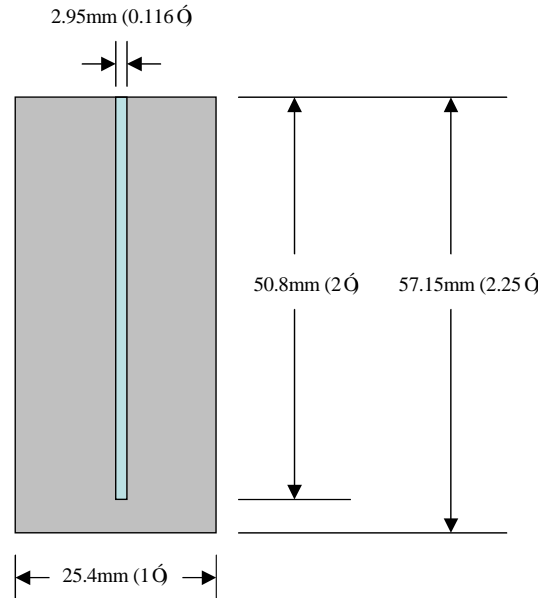
Next, the gray level, G_U , of every voxel containing an unknown fractional water mass, m_U , is normalized by the gray level, G_R , of a voxel with a known water mass, m_R , then the unknown fractional amount of water mass can be determined via:

$$m_U = \frac{G_U}{G_R} * m_R$$

This normalization can be performed because in NCT a linear dependence between a voxel’s gray level and the total macroscopic cross section of the material it represents is expected [4], thus ensuring the resulting fractional value of a voxel gray level reflects the same fractional amount of water with respect to the known water mass. The total water mass may be found by summing the individual voxel water masses. In practice, the gray level of a voxel known to be filled with water is chosen for the reference gray level G_R .

2.2.1 Experimental Setup and Results

The NCT water quantification method outlined above was tested using an aluminum cylinder test sample shown in Fig. 7 and the NCT system installed in the first 2 years of this program. The cavity of the test sample was drilled with a #32 drill bit. – Kevin, please change all bit size references to diameter references. This is the only confusing part.



Is the diameter symbol here messed up?

Fig. 7. Dimensions of aluminum cylinder test sample.

Two projection data sets were taken, one of the sample empty and one of the sample filled with water (for background normalization purposes). Each data set contained 601 images acquired at 0.3° intervals. All images were taken with an integration time of 49s and at a reactor power of 800kW.

Image analysis was performed on a dedicated data processing computer utilizing the neutron and x-ray tomography reconstruction software, Octopus V8.2, the 3-D visualization program, VG Studio Max 1.2 (both acquired during the first year of this program), and the 2-dimensional water quantification program, PSUMagic [5] (developed in-house prior to the first year of this program).

As described in the technique synopsis above, each image in the projection data set of the sample filled with water was background normalized using its corresponding image from the projection data set of the dry sample before reconstruction. An example of the projection data before and after background normalization can be seen in Fig. 8. Fig. 9 shows the 3-D volume reconstruction of the background normalized water column.

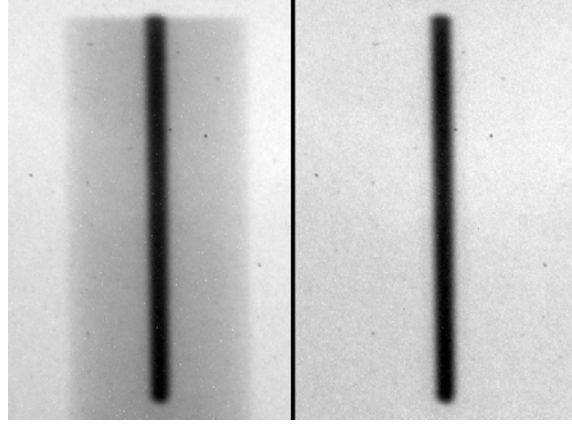


Fig. 8. Example of projection data before (left) and after (right) background normalization.

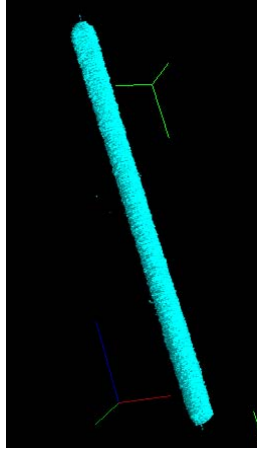


Fig. 9. Reconstruction of background normalized water column.

The reconstruction was then analyzed using the quantification method described above. Recall a reference gray level, G_R , is needed. Voxels of fractional water mass are likely to occur along the outer edge of the water column, but not within the core. The reference gray level was taken to be the average gray level of the water column's interior voxels. This was determined by averaging the interior voxel's gray levels in the x, y and z directions. After normalizing to the reference gray level, the water mass was calculated on a voxel-by-voxel basis; multiplying each voxel by the reference voxel's water mass, $(0.113 \text{ mm})^3 \times 1 \text{ mg/mm}^3$, where a water density of 1 mg/mm^3 is assumed.

For comparison, the water present in the test sample was quantified using a 2-dimensional radiosopic image and PSUMagic [5]. A theoretical water mass was determined using the test sample void dimension and assuming a water density of 1 mg/mm^3 . In addition, VG Studio Max was used to calculate the water mass of the reconstructed column (including and excluding partially filled voxels) based on the size of a voxel alone and not the amount of water mass it represents. This was done to demonstrate the importance of both including partially filled voxels and accurately determining the water mass they represent. These results may be found in Table 1.

Table 1. Results of Water Mass Analysis

Method	Water Mass (mg)	Error (%)
Reference Gray Level G_R	350.8	1.3
PSUMagic	346.0	-0.1
Theoretical	346.4	NA
VGStudio Max w/o Fractional	311.6	-10
VGStudio Max w/ Fractional	398.2	15

The comparison of the results found in Table 1 reveals the quantification of water mass using a reference gray level is a viable approach with accurate results. It also reveals the importance of including partially filled voxels and calculating their amount of water mass. Failing to do so will yield largely inaccurate results.

2.3 Quantification of Ice in Simulated Fuel Cell Flow Fields

To demonstrate the ability to quantify ice, several additional test samples were constructed in addition to the one from the previous section. #29, #22, and #16 drill sizes were used in making the cavity diameters of the additional test samples 0.136" (3.454mm), 0.157" (3.988), and 0.177" (4.496), respectively. [Change the drill bit refs here and below](#)

Each of the test samples was chilled to sub-zero temperatures for the duration of projection acquisition through the use of dry ice. A known amount of water, measured via syringe, was quickly "injected" into each test sample's cavity where it promptly froze. The amount of water injected was always less than the sample cavity's total volume to allow for expansion of the water during freezing. Fig. 10 shows projections of each of the samples filled with ice.

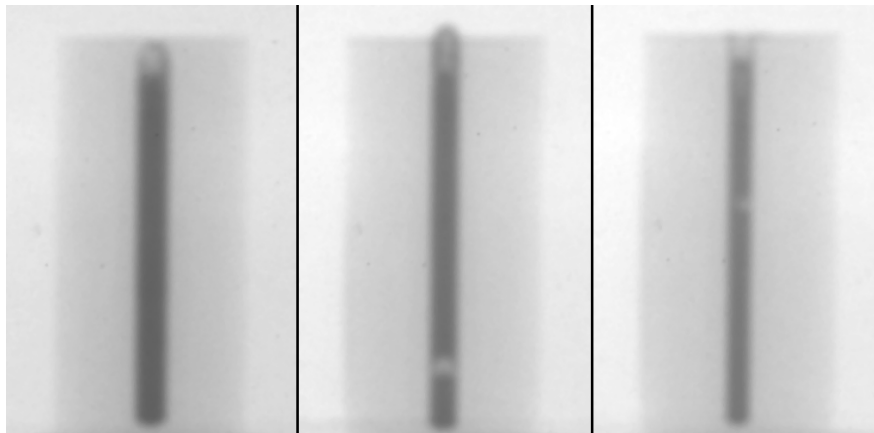


Fig. 10 Example projections of the test samples before background normalization. Drill sizes from left to right: #16, #22, and #29. [change bit refs](#)

The methodology of Section 2.2.1 was then applied to each test sample's projection data to perform ice quantification. The results of these analyses can be found in Table 2.

To further demonstrate water and ice quantification capabilities, test sample #32 was imaged with the same amount of water both frozen and thawed. Both sets of projection data were then analyzed and the water mass and ice mass quantified. Fig. 11 shows projections of the test sample with cavity drill size #32 frozen and thawed and Table 2 presents the quantification results.

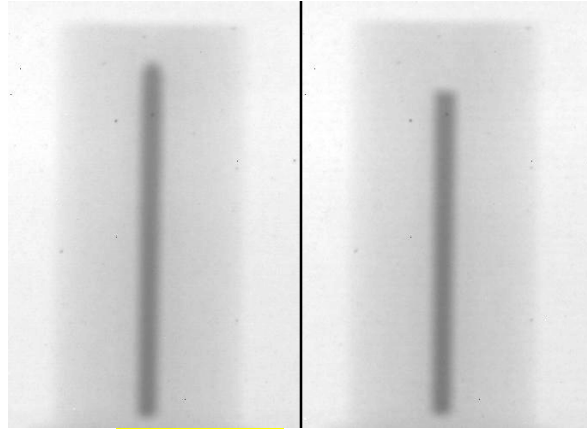


Fig 11. Test sample of cavity drill size #32 with the same amount of water frozen (left) and thawed (right). Note the change in water column height due to ice expansion.

Table 2. Results of Ice or Water Mass Analysis.

Drill Size and Condition	Diameter (mm)	Theoretical Mass (mg)	Measured Mass (mg)	Error (%)
#16 Frozen	4.496	700	705.8	0.8
#22 Frozen	3.988	600	588.3	-1.9
#29 Frozen	3.454	400	388.1	-3
#32 Frozen	2.946	300	298.0	-0.7
#32 Thawed	2.946	300	295.3	-1.6

The cavity of each test sample can be interpreted as a portion of a fuel cell flow field: a small channel blocked with ice. Table 2 shows that quantification of ice inside a small channel are accurate to within 3%. Thus, the ability to quantify ice within a fuel cell flow field under cold-start conditions was demonstrated under realistic length scales relevant to fuel cells.

2.4 Quantification of Ice Water in Simulated Fuel Cell Flow Field – Objective #3 Completion

While it is important to be able to quantify liquid water and also to be able to quantify ice, there is a potential that the two may coexist with each other during the startup phase of a frozen fuel cell. In this situation it may not be possible to fully distinguish between liquid and ice phases within an ice water mixture. This raises concerns over how to

appropriately quantify the mixture. One could conceivably determine the gray level value of a voxel within the volumetric reconstruction that completely represents ice and then determine another that completely represents water, but this could prove difficult. It also still **wrestles (got to be a better word than wrestles)** with determining which voxels to be analyzed are water and which are ice.

To solve this issue, we have determined that it is possible to use the gray level of a voxel that completely represents water to quantify both ice and water in a mixture. As water freezes, the density also changes. Therefore, the voxels that are used to represent ice can be interpreted as partially filled water voxels. In addition, as water freezes it also expands. In this sense a mass of water represented by a number of filled voxels can be interpreted as the same mass of water occupying a larger number of partially filled voxels when it turns into ice. As a result, normalizing volumetric reconstruction data set of an ice water mixture with the method outlined in Section 2.2.1 should result in an accurate quantification result.

To test this approach, the reconstructed data of the frozen test sample with cavity drill size #32 was normalized using the gray level value of a voxel representing water. The value of the voxel came from the data of the sample thawed. The renormalized data set was then quantified. The result may be seen in Table 3 alongside the quantification results for the test sample from Table 2, for comparison.

Table 3. Analysis of Test Sample with Cavity Drill Size #32

Drill Size and Condition	Diameter (mm)	Theoretical Ice Mass (mg)	Measured Ice Mass (mg)	Error (%)
#32 Frozen (Normalized to Ice)	2.946	300	298.0	-0.7
#32 Thawed (Normalized to Water)	2.946	300	295.3	-1.6
#32 Frozen (Normalized to Water)	2.946	300	296.1	-1.3

As can be seen in the final row of Table 3, analyzing the frozen sample using a normalization value obtained from the thawed sample yields an accurate (within 1.3%) quantification result. This demonstrates it is possible to quantify both water and ice using a voxel that is representative of water alone. This also demonstrates how quantification can be performed in situations where it may not be known at all if there is water, or ice, or a mixture present, which is appropriate for a fuel cell during cold start-up or shut-down situations.

3. Summary

The overall three year NEER program at Penn State has been successful, and all major research objectives have been achieved. Major new enabling systems for enhanced image and reconstruction quality have been installed and verified. A water quantification technique for NCT was developed and tested. The ability to quantify water, ice and ice water mixtures in NCT data sets has been demonstrated. Following the three years of

progress, a high-resolution imaging system, reconstruction software, and quantification software are now in place, and the ability to visualize and quantify multi-phase mixtures of ice and water with high precision in operating fuel cells has been demonstrated. The academic achievements of the project are also noteworthy, and are summarized in Sections 5 and 6 of this report.

4. Planned work

All of the planned objectives of the project have now been met. The new system at Penn State developed through this project will be used to attract new external research contracts and provide the fuel cell and other science communities with a new tool to assist in design, development, and understanding.

5. Student Theses Generated During the Project at Penn State

B.S. with Honors Degrees in Mechanical Engineering

(2006) Stephen Soung, Method for Freezing Fuel Cells for Neutron Imaging

(2006) Christopher Somers, Development of Test System to Image Frozen Water in a Fuel Cell in 3-D

Master of Science Degree in Nuclear Engineering

(2008) Liang Shi, Development of a Neutron Computed Tomography System at The Pennsylvania State University (leveraged with Big10 INIE funds)

Ph.D. Degree in Nuclear Engineering

(Sp 2009 Anticipated) Kevin Heller, Water Quantification Techniques for Neutron Computed Tomography

(Sp 2010 Anticipated) Liang Shi, Neutron Computed Tomography, Neutron Computed Tomography Water and Ice Characterization (leveraged with Big10 INIE funds)

(Sp 2009 Anticipated) Ahmet Turhan, The Nature of Two-phase Flow Phenomena in PEFCs (Leveraged with industrial funding)

6. Publications and presentations to date

Archival Journal Publications and Refereed Proceedings Resulting from Complete or Partial Leveraged Funding from this Project

Cho, K. T., Turhan, A., Lee, J. H., Brenizer, J. S., Heller, A. K., Shi, L. and Mench, M. M. 2008. Probing Water Transport in Polymer Electrolyte Fuel Cells with Neutron Radiography, *in press, Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment in press.*

Heller, A. K., Shi, L., Brenizer, J. S., and Mench, M. M. 2008. Initial Water Quantification Results using Neutron Computed Tomography, *in press, Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment in press.*

Turhan, A., Heller, K., Brenizer, J. S., and Mench, M. M. 2008. Passive Control of Liquid Water Storage and Distribution in a PEFC through Flow Field Design, *Journal of Power Sources, Volume 180, Issue 2, 1 June 2008, Pages 773-783.*

Turhan, A., Heller, K., Brenizer, J. S., and Mench, M. M. 2006. Quantification of Liquid Water Accumulation and Distribution in a Polymer Electrolyte Fuel Cell Using Neutron Imaging, *Journal of Power Sources*, **160**, pp. 1195-1203.

Kowal, J. J., Turhan, A., Heller, K., Brenizer, J. S., and Mench, M. M. 2006. Liquid Water Storage, Distribution, and Removal from Diffusion Media in PEFCs. *Journal of Electrochemical Society*, **153**, pp. A1971-A1978.

Heller, A. K., Shi, L., Brenizer, J., and Mench, M. M. 2008. Error Analysis of Water Quantification Using Neutron Imaging, Neutron Radiography: *Proceedings of the 8th World Conference on Neutron Radiography (WCNR-8)*, pp 134-145 (2008).

Turhan, A., Kowal, J. J., Heller, K., Brenizer, J., and Mench, M. M. 2006. Diffusion Media and Interfacial Effects on Fluid Storage and Transport in Fuel Cell Porous Media and Flow Channels. *ECS Transactions*, **3**, 1, *Proton Exchange Membrane Fuel Cells 6*, Eds. T. Fuller, C. Bock, S. Cleghorn, H. Gasteiger, T. Jarvi, M. Mathias, M. Murthy, T. Nguyen, V. Ramani, E. Stuve, T. Zawodzinski, pp. 435-444.

Presentations

Heller, A. K., Shi, L., Brenizer, J., and Mench, M. M. 2008. Initial Water Quantification Results Using Neutron Computed Tomography, *presented at The 6th International Topical Meeting on Neutron Radiography (ITMNR-6)*. Kyoto, Japan, September 15-19, 2008.

Shi, L., Heller, A. K., Brenizer, J., and Mench, M. M. 2008. Development of a Neutron Computed Tomography System at Penn State University, *presented at The 6th International Topical Meeting on Neutron Radiography (ITMNR-6)*. Kyoto, Japan, September 15-19, 2008.

Cho K., Lee, J. H., Turhan, A., Heller, A. K., Shi, L. Brenizer, J. S., and Mench, M. M. 2008. Probing Water Transport in Polymer Electrolyte Fuel Cells with Neutron Radiography, *presented at The 6th International Topical Meeting on Neutron Radiography (ITMNR-6)*. Kyoto, Japan, September 15-19, 2008.

Turhan, A., Kowal, J. J., Heller, K., Shi, L., Brenizer, J., and Mench, M. M. 2006. Interaction of Design, Materials, and Interfacial Forces on Liquid Water Storage and Distribution in Polymer Electrolyte Fuel Cells, *presented at the 8th World Conference on Neutron Radiography (WCNR-8)*. NIST, October 16 - 19, 2006.

Heller, K., Shi, L., Brenizer, J., and Mench, M. M. 2006. Error Analysis of Water Quantification Using Neutron Imaging, *presented at the 8th World Conference on Neutron Radiography (WCNR-8)*. NIST, October 16 - 19, 2006.

Shi, L., Heller, K., Brenizer, J., and Mench, M. M. 2006. The Penn State University Neutron Computed Tomography Facility. *presented at the 8th World Conference on Neutron Radiography (WCNR-8)*. NIST, October 16 - 19, 2006.

Mench, M. M., Turhan, A., Keller, K., P. A. Chuang, Ünlü, K., and Brenizer, J. 2005. INIE Big-10 Consortium Enabled Research: A new Physical Model of Two-Phase Transport in Polymer Electrolyte Fuel Cells Using Neutron Imaging at Penn State. *Presented at the Fall American Nuclear Society Meeting*, November, 2005.

7. References

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- [2] D. Kramer, J. Zhang, R. Shimo, E. Lehmann, A. Wokaun, K. Shinohara, and G. G. Sherrer, "In Situ Diagnostic of Two-Phase Flow in Polymer Electrolyte Fuel Cells by Neutron Imaging Part A. Experimental, Data Treatment, and Quantification." *Electrochimica Acta* **50**, Issue 13, pp. 2603-2614 (2005).
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- [4] M. Zanarini, P. Chirco, M. Rossi, G. Baldazzi, G. Guidi, E. Querzola, M. G. Scannavini and F. Casali, "Evaluation of Hydrogen Content in Metallic Samples by Neutron Computed Tomography," IEEE Transaction on Nuclear Science, Vol. 42, No. 4, pp. 580-584, (1995).
- [5] A. K. Heller, P. A. Chuang, J. Brenizer, K. Ünlü, "Water Quantification Using Neutron Imaging," Transactions of the American Nuclear Society 93, pg. 860-861 (2005).