AN ABSTRACT OF THE THESIS OF

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Title: Characterizing Accelerated Weathering Conditions for Wood Adhesive Bonds

Abstract approved:

Frederick A. Kamke

Accelerated weathering (AW) tests have become a requirement for many wood composite products. This project monitored temperature and moisture content of specimens undergoing AW exposure for the following standards: ASTM D3434 (automatic boil test), CSA O112.9 (boil-dry-freeze), and PS2 (Section 7.17, 6-cycle VPS). Each AW procedure was conducted with three types of specimens that differed by geometry and size. Using fine-wire thermocouples, temperature was measured in the center of the specimen during AW regimes. Average MC of specimens was determined by weight measurements, along with a final oven-dry weight. MC gradient was determined by periodic destructive testing, by removing a specimen from the AW environment and promptly cutting into sections for a gravimetric MC determination. Results include plots of temperature and MC as a function of time and/or cycle. Mechanical tests were performed on the weathered specimens, as well as dry control specimens.

Geometries included lap-shear in tension, compression shear blocks and short-span shear in bending (SSB) which was manufactured using laminated veneer lumber (LVL). Direct comparisons between AW methods were

conducted through non-linear regression by fitting the VPS and BDF data to equivalent cycles of the ABT. The SSB specimens showed to retain the greatest amount of strength after mechanical testing while the compression-shear block geometry had the greatest overall loss in strength. Lap-shear specimen mechanical results yielded the greatest variability among all three AW regimes. Temperature measurements were as expected within the lap-shear and compression block geometries, indicating that they were receptive to the exposure conditions. The SSB geometry, showed to be the least responsive to changes in temperature throughout exposure conditions in ASTM D3434.

MC measurements, taken after each test's respective drying phase, showed to be under the fiber-saturation point within CSA 0112.9 and PS2 6-cycle vacuum pressure soak test, as expected. Results of ASTM D3434 showed that there was essentially no occurrence of drying among all three specimen geometries.

Characterizing Accelerated Weathering Conditions for Wood Adhesive Bonds

by

Micah L. Sutfin

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APPROVED:

Major Professor, representing Wood Science

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I understand that my thesis will become part of the permanent collection of Oregon State University libraries. My signature below authorizes release of my thesis to any reader upon request.

Micah L. Sutfin, Author

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LIST OF ABBREVATIONS

ABT: Automatic Boil Tester

APA: The Engineered Wood Association

ASTM: American Society for Testing and Materials

AWG: American Wire Gauge

BL: Bondline

BDF: Boil - Dry - Freeze

C: Compression block specimen

CPU: Compression block specimen with Polyurethane

CPF: Compression block specimen with Polyurethane

CSA: Canadian Standards Association

DC: Dry control

EPI: Emulsion polymeric isocyanate

FSP: Fiber saturation point

LVL: Laminated veneer lumber

MC: Moisture content

NIST: National Institution for Standards and Technology

OD: Oven Dry

PB: Particleboard

PF: Phenol-formaldehyde

PWF: Percent wood failure

PU: Polyurethane

PS2: Product Standard 2

RH: Relative humidity

SSB: Short-span shear in bending specimen

T: Lap-shear tension specimen

TPF: Tension phenol formaldehyde

VPS: Vacuum Pressure Soak

WC: Wet control

DEDICATION

I wish to dedicate this work to my late father, David Lee Sutfin. Growing up, my father was a carpenter, woodworker and a master of his trade, eventually working his way to a project management position for a residential builder. It was my father that taught me the virtue of a 2x4 and the value of a finished product. As a young adult, my brothers, my sister and I would spend our summers building decks and other wooden-accessory structures with our Father. I soon fell in love with buildings. When my father passed away in 2011, my respect for wood increased tenfold.

1. Introduction

Building codes regularly specify that engineered wood products (EPWs) conform to individual product standards. Product standards provide the configuration for EWPs and specify elements such as product use limitations, material specifications, engineering properties and quality control measures. Adhesives employed in EWPs must meet all requirements specified in an individual product standard in order for the product to comply with the references in the building code.

Product and adhesive standards are generally published and reviewed by standards agencies such as ASTM and CSA. When standardized adhesive test method is specified, instructions for qualification will be listed in the product standard. Numerous third party agencies and testing laboratories are involved qualification process to observe specified tests that are to be conducted. Adhesive standards utilize methods of accelerated weathering to conduct durability assessments for adhesives employed in EWPs.

Often methods of accelerated weathering are intended to differentiate between the exterior durability of two or more adhesives, coatings and other treatments employed in EWPs. AW procedures have become the means for generating information about durability, which is defined as the capability of maintaining the serviceability of a product, component assembly or construction over a specified period of time (ASTM E 632).

AW procedures depend on the standard test method chosen. Conditions may involve room temperature water soaking, hot water soaking, vacuum pressure soaking, submersion in boiling water, water spray, convection air drying, freezing, ultraviolet light or some form of outside exposure to the weather.

These tests subject the adhesive bonds in these products to varying rate and extent of temperature and moisture content change. While there are few claims that AW testing provides data that can be used to predict service life, AW testing is useful as a comparative test among product alternatives.

AW tests have become a requirement for many wood composite products. The goal of this project was to characterize the bondline conditions of test specimens undergoing standard accelerated weathering procedures. This project monitored temperature and moisture content of specimens undergoing AW exposure for the following standards: ASTM D3434 (automatic boil test), CSA O112.9 (boil-dry-freeze), and PS2, section 7.17, (6-cycle VPS).

Each AW procedure was conducted with three types of specimens that differed by geometry and size. Using fine-wire thermocouples, temperature was measured in the geometric center of the specimens during AW regimes. Average MC of specimens was determined by weight measurements, along with a final oven-dry weight. MC gradient was determined by periodic destructive testing, by removing a specimen from the AW environment and promptly cutting into sections for a gravimetric MC determination. Results include plots of temperature and MC as a function of time and/or cycle. Mechanical tests were performed on the weathered specimens, as well as dry control specimens.

2. Literature Review

2.1 Effect of Specimen Size

The influence of specimen size should be considered for all durability and mechanical testing, for not only wood products, but all structural materials. Specimen size can affect the ingress and egress of moisture in the form of liquid and vapor. Moisture ingress can be significantly higher along structural wood panel edges, where wood's transverse plane provides less resistance into the panel than that of the tangential and radial surfaces (Way et al., 2019). The authors claimed that edge effects in smaller specimens are likely to accelerate degradation, while larger specimens tend to experience less. This is likely accounted for the relatively smaller volume, increasing the rate of moisture sorption throughout the material.

Kojima (et al. 2017) compared the retention of MOE, MOR, and internal bond strength (IB) of structural grade particleboard when exposed to outdoor conditions. Two specimen dimensions were included in the study – 300 x 300 mm^2 and 300 x 150 mm^2. They found that MOR and MOE retention for the smaller rectangular specimens was about 3 percent lower than for the larger square specimens, and the IB retention for the smaller specimens was about 7 percent lower than the larger specimens. Both of these studies would indicate that specimen size could be correlated with their realized mechanical and hygroscopic attributes.

2.2 ASTM D-3434(2018) - Automatic Boil Test

Many in the wood products industry are skeptical to the concept of boiling engineered wood products such as LVL and plywood. While this may seem peculiar, wood does absorb water which boiling accelerates. ASTM D3434-00

Standard Test Method for Multiple-Cycle Accelerated Aging Test (Automatic Boil Test) for Exterior Wet Use Wood Adhesives is an aggressive test procedure that has fallen out of favor due to the complex and specialized nature of the apparatus (ASTM D3434, 2018). While this machine was commonly used by the Weyerhaeuser company for durability testing, the sole automatic boil tester known to exist in was recently donated to Oregon State University. ASTM D3434 is a rather unique standard among AW procedures for engineered wood products. The standard is distinctive, in that it is an automated procedure.

Additionally, the ASTM D3434 requires mechanical testing on specimens at selected cycles, which ultimately is intended to yield a bondline strength-retention curve. ASTM D3434 has been an active standard since the mid-1970's and has been renewed as of 2018. The ABT, as mentioned, is an automated test. Because it is automated, the standard requires a special machine to facilitate the procedure. A diagram of the automatic boil tester can be seen in Figure 2-1.

There are 1600 total cycles in the standard, each cycle consisting of three phases: wet, cool and dry. One accelerated weathering cycle includes 10 min boiling water, 4 min in 23°C ± 2°C air and 57 min in 107°C ±2°C air. US Product Standard ANSI 405 has found significant loss in bondline strength retention after 200 cycles (APA, 2011). Because of this, most researchers have typically adhered to 800 cycles, opposed to 1600 specified in the standard.

ASTM D3434 is specified in ANSI 405 Standard for Adhesives for Use in Structural Glued Laminated Timber. ANSI 405 is also referenced by ANSI PRG 320-2012 Standard for Performance-rated Cross-laminated Timber, however, ASTM D3434 is excluded for adhesive qualification. ANSI 405, section C405.2.1.5 states "*While the automatic boil test in ASTM D3434 is considered an excellent test, limited accessibility of test equipment makes an alternative test desirable. Limited data suggest that CSA 0112.9 provides a useful alternative to ASTM D3434*."

ASTM D3434 is an accelerated weathering (AW) method for assessing the durability of wood adhesive systems that may be suitable for exterior or wet-use applications. The automatic boil test refers to a device called Automatic Boil Tester (ABT), which subjects test specimens to cyclic conditions of boiling water, cool air and hot air.

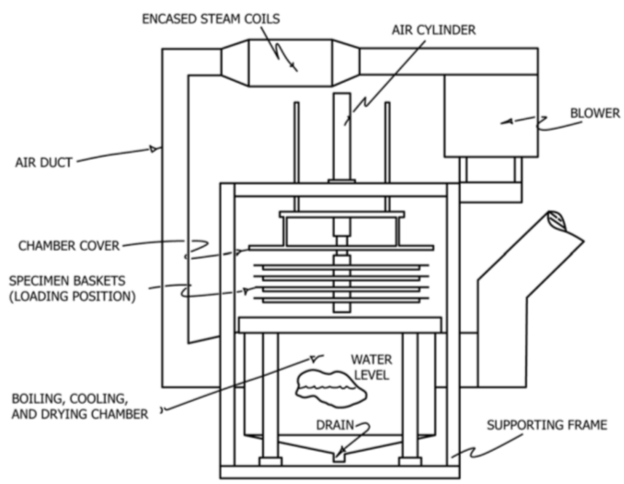

Figure 2-1, Automatic Boil Tester (ASTM D3434-00)

This test was developed by Northcott et al (1968) in effort to account for a method of AW that would correlate with service life of plywood and laminated timber. What they found was cycles consisting of 10-min in boiling water, followed by specimens being submerged in ice-cold water for 3.75 min, followed by 60-min of drying at 225°F was the most promising of 11 test alternatives. This ultimately led to the development of ASTM D-3434, differing in that the "ice" phase was replaced with "cool phase".

Drying is considered in respect to wood's fiber saturation point (FSP). FSP is used to designate the point in the drying process at which only water bound in the cell walls remains and liquid water, called free water, having been removed from the cell cavities. When wood is below FSP, dimensional changes occur. The FSP of wood varies among species, but is generally considered to be approximately 30% MC (Simpson, 1991).

Previous reports by Wilkie (1974) indicate very little drying stresses in the specimens subjected to the ABT exposure. The configuration of the test specimen shows the optimum stress occur between boiling and drying, but insignificant checking in the wood was observed. This could perhaps be due to the lack of air and water circulation between the specimens. Wilkie bored a 0.25 inch hole in the lap-shear specimens and placed a metal rod and spacers between them. This was done to mitigate inadequate air/water circulation. The standard does not specify this to be done, but may prove to be useful in future if specimens are dried inadequately.

2.3 CSA 0112.9 – Boil-Dry-Freeze Test

CSA O112.9 is another AW regime intended to evaluate adhesives used to bond wood for possible exterior applications. While the BDF test is not as arduous in nature as the ABT, it remains useful as most steps can be accomplished with basic laboratory equipment, such as a convection oven and a boiling water vat (CSA 2009). The BDF test subjects specimens to cyclic conditions of boiling, drying and freezing. While this test method is different in its cyclic nature, it is also a substitute for the ASTM D3434 according to ANSI 405. The primary difference being the presence of the freezing phase as well as the length of boiling and drying phases being substantially longer. While freezing could be considered similar to the 'cool' phase of the ABT, it does not directly follow the boiling phase.

Some studies have shown that freezing wood-based composites induces stress in the material. Chow and Steiner (1974) aged exterior-grade plywood in subzero conditions and found much wood tissue damage promoted by severe internal stresses. In contrast, it was found that after removing the freezing phase from ASTM D1037, there was no significantly recognized difference of the final results (USDA 2016).

2.4 PS2 7.17 – 6-Cycle Vacuum Pressure Soak

The 6-cycle vacuum pressure soak test specified in Voluntary Product Standard PS2-18 is used to evaluate delamination and bondline strength retention for products rated as Exposure 1. PS2 states that "*Panels classified as Exposure 1 are intended to resist the effects of moisture on structural performance due to active construction*." Test specimens are subjected to 30 min in 66 °C water with a vacuum of 50.6 kPa (15 in. Hg) followed by 30 min in 66 °C water at ambient pressure, and then dried at 82 °C for 6hrs.

Steps 1 and 2 are then repeated and then dried at 82 °C for 15hrs. This completes two cycles of the test. Four additional cycles are repeated; then the specimens are mechanically tested when dry. Voluntary product standards, like PS2, provide rigid methods for AW procedures to assess relative moisture resistance, typically on a pass/fail basis (APA 2011). Pass/fail, in this case is an observance, and is based on a combination of wood/adhesive failure and bondline delamination.

3. Materials & Methods

This project monitored temperature and moisture content of specimens undergoing AW exposure for the following standards: ASTM D3434 (automatic boil test), CSA O112.9 (boil-dry-freeze), PS2 (Section 7.17, 6-cycle VPS). Each AW procedure was conducted with three different specimen geometries, being single-lap shear in tension, single-lap shear in compression and short-span shear in bending of LVL. These specimen geometries and associated mechanical test methods focus on the performance of the adhesive bond. The strategy for specimen selection was taken from actual specimen requirements specified by the standards.

Using fine-wire thermocouples, temperature was measured in the center of the specimen during each AW exposure. Average MC was determined by periodic weight measurements, and then a final oven-dry weight. MC gradient was realized by periodic destructive testing, by removing a specimen from the AW environment and quickly cutting into sections for a gravimetric MC determination. Results include plots of temperature and MC as a function of time and/or cycle. Appropriate mechanical tests were performed on the weathered specimens, as well as unweathered control specimens.

3.1 Specimen Geometry

This study utilized three separate specimen geometries. Geometries included were single lap-shear in tension (T), single lap-shear compression block (C) and short-span shear in bending (SSB), as described in ASTM D2339, D905 and D5456 respectively. The short-span shear specimen was 1.5” thick Douglas-fir, LVL provided by Boise Cascade (White City, Oregon) measuring 10.5 inch in length and 1.5 inch in width. The LVL was produced from two layers of phenol-formaldehyde-bonded LVL that was laminated together using emulsion polymer isocyanate (EPI); this is considered to be the primary bondline (PBL) of interest in the study. A depiction of the three specimen geometries can be observed in Figure 3-1. Note that specimens are not to scale.

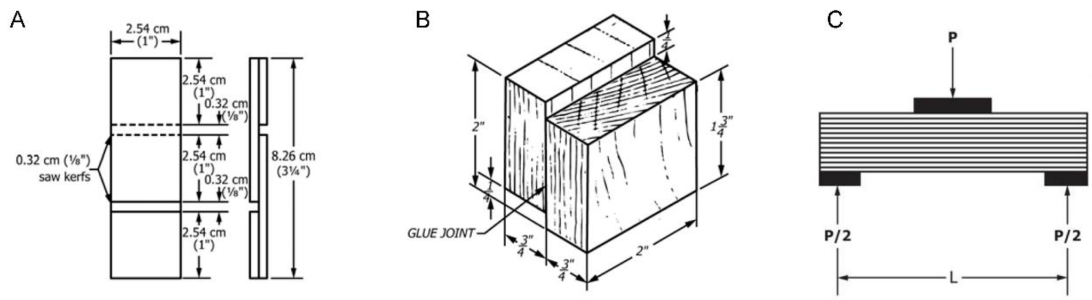

Figure 3-1, Specimen geometry; A) ASTM D2339 single lap-shear in tension (T), B) ASTM D905 single lap-shear in compression (C), C) ASTM D5456 short-span shear in bending (SBB)

All specimens were manufactured using Douglas-fir (*Pseudotsuga menziesii*) heartwood with a very narrow density range of 480-520 kg/m^3, consistent with the average density of the species (USDA, 2010, Ch.5). Specimen sizes and geometries were selected because they are commonly applied in durability tests for EWPs. Furthermore, each geometry varied greatly in size, which will provide insight as to how each AW condition effects the sample undergoing exposure. Table 3-1 illustrates the relationship between specimen geometry, adhesive type and their respective nomenclature.

Table 3-1, Specimen Nomenclature

Geometry	Adhesive Type	Nomenclature
T	Phenol formaldehyde	TPF
C	Phenol formaldehyde	CPF
C	Polyurethane	CPU
SSB	Emulsion Polymer Isocyanate (PBL)	SSB

3.1.1 Specimen Preparation – Lap-Shear (T) and Compression Block (C)

Lap-shear specimens were manufactured in accordance with ASTM D 2339. The adhesive employed in this geometry was phenol-formaldehyde, premixed for LVL application which was provided by Arclin (Springfield, Oregon). The solids content of this adhesive was approximately 49%. The Douglas-fir substrate was milled to approximately 0.3 inch and then stored in an environment room at 20°C and 65% RH for one week. Following this, the substrate was knife-planed to 0.25 inch, 24 hours prior to bonding.

The manufacturing of the PF single-lap shear in compression block specimens was similar to that of the lap-shear in tension specimens, only differing in how the specimens were cut after assembly. Additionally, because this geometry is substantially larger than the lap-shear in tension specimens, they were equilibrated in the standard room for two weeks instead of one, prior to planing.

The compression block sample group included two adhesives, the same PF as mentioned previously and polyurethane (PU). The PU adhesive was provided by the Henkel Corporation. This proprietary formula falls under the trade name Loctite HB X202 Purbond ™. A spread rate of 125 g/m^2 was used for this adhesive formulation. One lamina was coated with PU using a notched spreader while the other was left uncoated. The two lamellae were assembled and pressed at 150 psi for 60 min, using a 24 x 24 inch cold-platen press (Figure 3-2). The environmental conditions of the room were ~ 20 °C and 65% RH, consistent with the manufacturer's recommendation, using a 24x24" cold-platen press.

Figure 3-2, PU compression block assembly in cold press

PF adhesive was applied to one side of the bond line at a spread rate of 120 – 125 g/m^2 based on solids content (Figure 3-3). Again, a notched applicator was used to achieve even distribution of adhesive on the substrate. Both lamellae were assembled and pressure was applied by a hydraulic press at 125 psi and 170°C for 10 min for geometry T and 19.5 min for geometry C (Figure 3-3). Bond line temperature was monitored with a thermocouple, with a goal to achieve 100°C for at least 2 min. Pressure was slowly released and the sample removed from the hot-press. The bonded samples were stored at 20°C and 65% RH for one week, and then cut according to Figure 3-1.

Figure 3-3, PF sample preparation; single bondline (left) and hot-pressing in 6-inch by 6-inch electrically-heated press (right)

3.1.2 Specimen Preparation – Short-span Shear in Bending

The SSB sample group was provided by Boise Cascade (White City, Oregon), therefore required no preparation other than cutting them to the specified dimension in accordance with section A4 of ASTM D5456, short-span shear in bending specimen. As specified in A4 of ASTM D5456, the span-to-depth ratio of this geometry is 6:1. Because the thickness of the LVL was 1.75 inch, the overall length of the specimen was cut 10.5 inch, allowing for 0.5 inch of overhang on either side of the supports during the mechanical testing (Figure 3-1).

The LVL was comprised of two 0.75 inch laminas bonded together with EPI adhesive, forming the primary adhesive bondline of focus in this study. While the individual laminas were comprised of multiple 0.125 inch veneers, bonded with phenol formaldehyde, focus of strength retention and delamination results was aimed at the primary bondline. After both geometries were cut to their respective dimensions, they were stored for at least one week at 20°C and 65% RH prior to AW exposure. Note that the short-span shear test places the greatest horizontal shear stress on the EPI bond in the center plane of the specimen.

A visual depiction of the completed specimens can be seen below in Figure 3-4. It is important to note the difference in specimen volumes. The specimens measure 0.81 in^3, 5.25 in^3 and 26.25 in^3 respectively for the T, C and SSB geometries. This represents a significant difference in relative specimen volumes. In addition, the half-thickness (distance from top and bottom surface to the bond line) was 0.25 inch, 0.75 inch and 0.75 inch for T, C, and SSB geometries, respectively.

Figure 3-4*, Completed lap-shear in tension specimen (left), lap-shear in compression specimen (center) and short-span shear in bending specimen (right).*

3.2 Accelerated Weathering Regimes

The AW methods chosen for this study include, ASTM D3434 - 00, CSA O112.9 – 10, and PS2 - 18 section 7.17. These methods were chosen for their differing AW conditions. This approach allowed a differentiation for each specimen geometry affected by the extreme conditions. The cyclic conditions of each AW method are as follows:

ASTM D3434 – Automatic Boil Test

1. Boil 10 min
2. Cool at 23°C, 4 min
3. Dry at 107°C, 57 min
4. Repeat steps 1-3 for 400 cycles
5. Boil 10 min, cool in water

CSA 0112.9 – Boil-Dry-Freeze

1. Boil *at least* 4hrs
2. Oven dry at 60°C for 19hrs
3. Freeze at ≤ -30°C for *at least* 4hrs
4. Repeat steps 1-3 for 8 total cycles
5. Boil 4 hrs, cool in water

PS2 7.17– 6-Cycle Vacuum Pressure Soak

1. 30 min in 66 °C water with a vacuum of 50.6 kPa (15 in. Hg)
2. 30 min in 66 °C water at ambient pressure
3. Dry at 82°C for 6hrs.
4. Repeat steps 1 and 2, then dry at 82°C for 15hrs.
5. This completes two cycles
6. Repeat 4 more cycles, for a total of six cycles

3.2.1 Dry and Wet Control Specimens

Dry control (DC) specimens were allowed to equilibrate in an environment chamber at 20°C and 65% RH for at least one week prior to mechanical testing. This was done to ensure reduce variability of MC, which greatly affects the mechanical properties of wood.

Wet control (WC) specimens are specified in ASTM D3434. WC specimens are required to be equilibrated prior to exposure. WC treatment includes, a 72hr. water soak test in ambient conditions. Specimens were placed in a wire-basket in groups of ten, in order to maximize water circulation. Following a 72hr. water soak, specimens were mechanically tested wet, in agreement with ASTM D3434.

3.2.2 Bondline Temperature Measurements

Temperature of the bondline was measured throughout cyclic exposure conditions using 30 AWG, type-k thermocouples placed in the center of each geometry, for each AW method. There were two replications. A single 0.125 inch hole was drilled directly into the center of each specimen. Following this, the thermocouple wire was placed into the hole along with a 0.125 inch poplar (*Liriodendron tulipifera*) dowel to account for any remaining void space.

After the thermocouple was in place, a high-temperature RTV silicone gasket material was applied to the top of the hole to eliminate moisture ingress. Thermocouple wires were soldered at the tip to mitigate electrical noise transmitted to the data acquisition software. A depiction of where the thermocouple was placed in each specimen can be observed in Figure 3-5. The location on the bondline, where the tip of wire was placed, is directly in the center of each specimen, which corresponds to the location of the primary bondline. After each thermocouple was installed, they were linked to a wireless transmitter which acquired data in ten-second intervals throughout each AW regime.

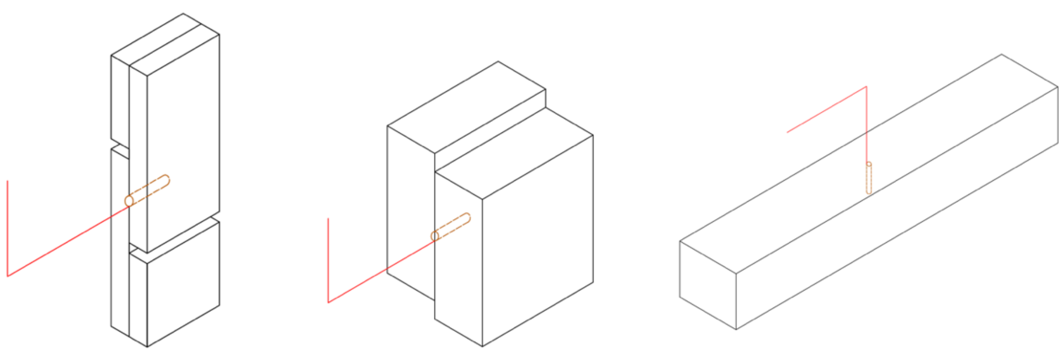

Figure 3-5, Thermocouple placement with dowel and wire location. From left to right: Specimen geometry T, C and SSB (not to scale)

3.2.3 Moisture Content Determination

Gravimetric MC measurements were taken from three specimens of each geometry, in accordance with ASTM D4442-20 *Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials*.

following each AW method's respective drying phase. This means that for ASTM D3434, measurements were recorded directly after the dry phase of cycles 20, 40, 100, 200 and 400. Similarly, measurements were taken after the dry phase of cycles 1-8 for the 8-cycle BDF test and cycles 1-6 for the 6-cycle VPS test.

Three specimens from geometries T, C and SSB were cut lengthwise into three segments of equal thickness, in order to isolate the bondline and two surfaces, which were averaged. After specimens were quickly segmented using a bandsaw, weight was recorded and then left to dry in a 103°C convection oven for 48 hrs. After specimens were allowed to dry, the segmented weights were recorded and averaged to determine average MC of the section. The two surfaces were averaged so they could be compared directly to the bondline MC. Figure 3-6 depicts how each specimen was segmented. The dashed lines represent the cut lines.

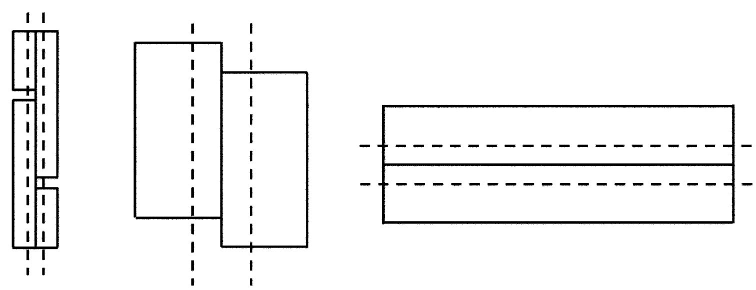

Figure 3-6, Segmented geometries for MC determinations. From left to right: Specimen geometry T, C and SSB

3.2.4 Experimental Design

Table 2 describes the experimental design chosen for this project. As mentioned previously three adhesive types PF, EPI and PU were employed in select geometries. For the T geometry, only PF was used in all three AW methods. For C, both PU and PF were used in ASTM D3434, but only PF was used in the BDF and VPS methods. Although the SSB includes PF, the EPI is the adhesive used on the primary bondline in this geometry and was utilized in all three AW regimes.

Table 3-2, Experimental design indicating the number of replications for each combination of AW treatment, specimen geometry, adhesive type, bondline temperature measurement, and moisture content measurement

TREATMENT	ADHESIVE	GEOMETRY			BL TEMP.			MC		
		T	C	SSB	T	C	SSB	T	C	SSB
ASTM D3434	PF	70	70		2	2		3/Cycle	3/Cycle	
	EPI			70			2			3/Cycle
	PU		70							
DC	PF,PU,EPI	30	30	30						
WC	PF,PU,EPI	30	30	30						
CSA O112.9	PF	30	30		2	2		3/Cycle	3/Cycle	
	EPI			30			2			3/Cycle
PS2 7.17	PF	30	30		2	2		3/Cycle	3/Cycle	
	EPI			30			2			3/Cycle

Thirty specimens of each geometry were tested as dry control (DC) samples. Similarly, 30 were tested for the wet control (WC) samples. All DC specimens were mechanically tested at approximately 12% MC in accordance with ASTM D3434. Furthermore, the DC sample groups were used to calculate percent strength retention of succeeding cycles, that required mechanical testing. For ASTM D3434 this includes cycles 20, 40, 100, 200 and 400. For the BDF and VPS tests, this includes only specimens from cycles 8 and 6, respectively. WC specimens were soaked in a temperature-regulated water bath for 72 hrs. at 25 °C, then mechanically tested wet according to the geometry’s respective mechanical test.

3.3 Destructive Testing

While specimen geometries were exposed to all three AW methods, their destructive testing was conducted in accordance to their required mechanical test. These will be discussed shortly. Specimens, regardless of geometry, were tested wet after D3434 and CSA 0112.9 exposure conditions. Specimens tested for the VPS exposure were tested when dry, in accordance with the standard. Percent wood failure was conducted in agreement with ASTM D 5266, *Standard Practice for Estimating the Percentage of Wood Failure in Adhesive Bonded Joints*. A supplementary adjunct, which is described in section 11 of D 5266, was also used for practicing the methodology and for ultimately determining percent wood failure of the T and C geometries. Due to the nature of the short span shear in bending test, percent wood failure was not accounted for in the SSB geometry group. However, the failure mode was recorded. All mechanical tests were performed on a universal testing machine.

3.3.1 Shear by Tension Loading

For group T, testing was performed in accordance with ASTM D2339, *Test Method for Strength Properties of Adhesives in Two-Ply Wood Construction in Shear by Tension Loading*. Specimens were weathered to their appropriate cycle and then tested at a rate of 0.157 inch/min in order to achieve a loading rate of 7.56 kg/s. A testing setup for ASTM D2339 can be observed in Figure 3-7. Specimens were tested until failure and the maximum load was recorded.

Figure 3-7, Mechanical test setup for T geometry in accordance with ASTM D 2339

3.3.2 Shear by Compression Loading

Geometry C was mechanically tested in agreement with ASTM D905, *Standard Test Method for Strength Properties in Shear by Compression Loading*. The loading rate was set to 4mm/min, as the maximum specified in the standard is 0.20 inch/min. The apparatus described in this standard is depicted in Figure 3-8. Again, specimens were tested until failure and the maximum load was recorded.

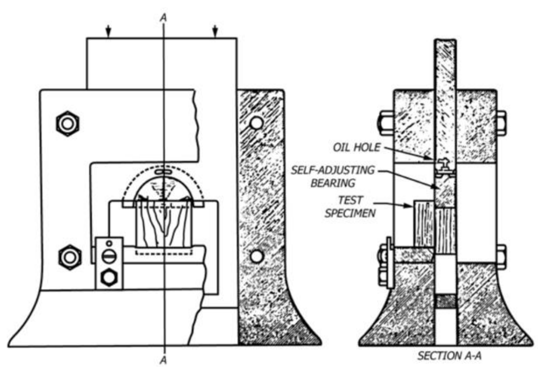

Figure 3-8, Mechanical test setup for C geometry in accordance with ASTM D905

3.3.3 Short-span Shear in Bending

The SSB group was tested according to section A4 of ASTM D5456, *Adhesive Durability Tests (Short Span Bending)*. A loading rate of 3.5mm/min was applied in order to observe failure within approximately 1 min. Specimens were tested until failure. The mode of failure, MC and maximum load was recorded. The testing configuration described in section A4 of ASTM D5456 can be observed in Figure 3-9.

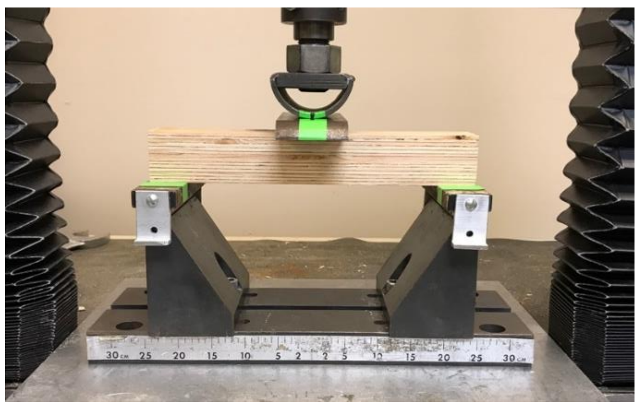

Figure 3-9, Mechanical test setup for SSB geometry in accordance with section A4 of ASTM D5456

3.3.4 The Automatic Boil Tester (ABT)

The Automatic Boil Tester (ABT) is composed mainly of a pneumatic cylinder, which moves the specimen chamber in and out of the boiling water, a blower, which provides the necessary airflow used in the cooling and drying phases, and a steam-heated heat exchanger to increase the air temperature Figure 3-10. The control system determines the time of each step in a cycle and the number of cycles desired.

ASTM D3434 has specific requirements regarding temperature and airflow rate between the steam heat exchanger and the sample chamber. Initially, the air temperature and flow rate were too low. An inlet air heater was added upstream from the blower fan. The control point temperature was established downstream from the blower fan, and controlled such that the inlet air temperature is maintained at approximately 30 °C. Air flow was increased by increasing the velocity of the blower fan.

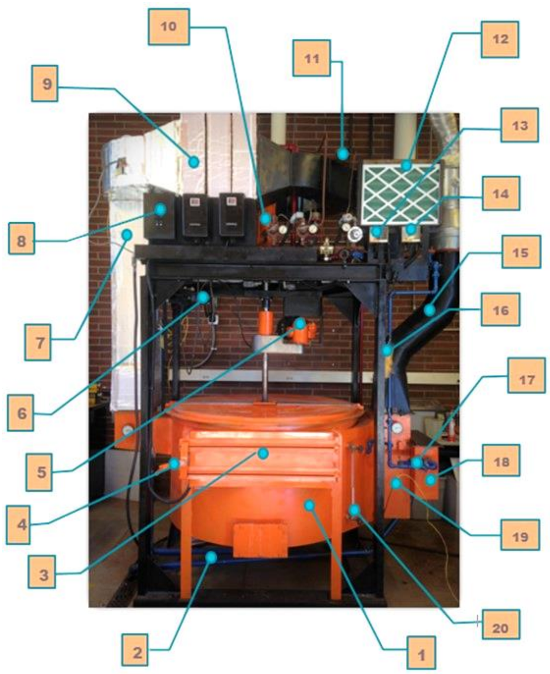

Figure 3-10, Automatic Boil Tester

<u>ABT Components</u>

1) ABT's chamber.
2) Drain line.
3) Access door.
4) Door actuator switch.
5) Rotation motor.
6) Rotation actuator switch.
7) Air duct.
8) ABT's circuit breakers.
9) Air heat exchanger.
10) Pneumatic cylinder.
11) Pneumatic damper.
12) Air dust filter.
13) Steam pressure switch.
14) Air pressure switch.
15) Exhaust steam duct.
16) Manual rotation controller.
17) Water line.
18) Water reservoir tank.
19) Thermocouple.

In order to have a record of the temperature, the operational times, and to calibrate the system, a data acquisition system was installed. The system is composed of a USB thermocouple unit, a device which is designed to operate with several types of thermocouples. The DAQ system provides graphical display in real time, and saves historical data records with various data formats. There are three type-k thermocouples, placed in the positions depicted in Figure 3-12. A thermocouple was also placed inside the water, which can be observed as component 19 Figure 3-10. This system offered assurance that the ABT's performance conditions were acceptable based on requirements of ASTM D3434.

Additionally, RH was estimated inside the ABT in accordance with the wet-bulb / dry-bulb method described by (USDA 2010, Ch.13). Two type-k thermocouples were inserted into an active cycle of the ABT, one with a wet cloth secured around the soldered tip, while the other remained bare. RH was estimated using an empirical equation derived by Stull (2011), so there is some error on the high and low end of the RH range. The DBT and WBT did not show to be stable. This could be due to rotation (3 RPM) of the basket, condensation of water dripping from the top of the ABT's chamber, or air turbulence.

Since DBT and WBT are not stable, the calculation of RH was also not stable. Sometimes the WBT was greater than DBT, which is not possible, and causes calculated RH to be greater than 100%. In those cases, RH was adjusted to 100%. This happened during the cooling phase and boiling phase. When the WB was submerged in the boiling water, RH was also adjusted to 100%.

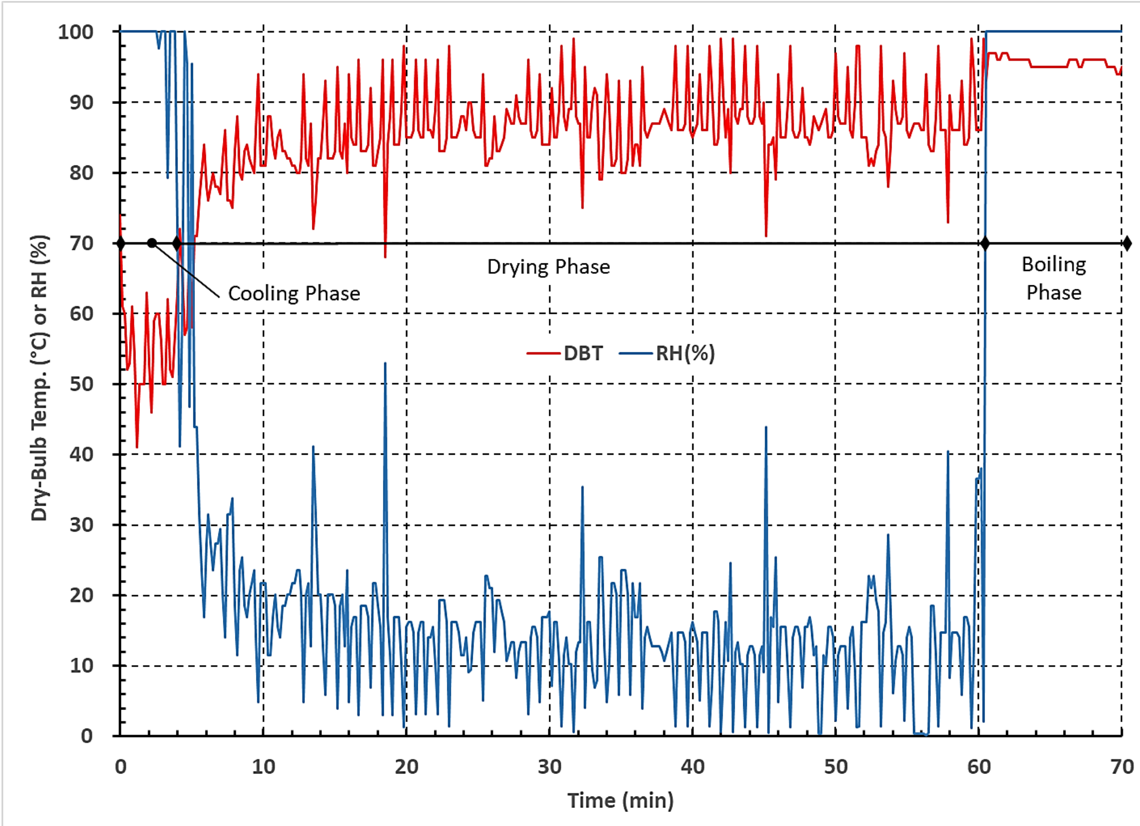

Figure 3-11, RH and Temperature conditions for 1 cycle, ABT

The data show that there was significant drying potential during the drying phase. The RH was about 10% for over 50 min (Figure 3-11). The temperature was lower than anticipated (~90 °C), but the EMC was still less than 2%. It can be concluded from this data that the specimens were subjected to adequate temperature and humidity to cause drying, but not uniformly on all surfaces of each specimen, nor for sufficient length of time.

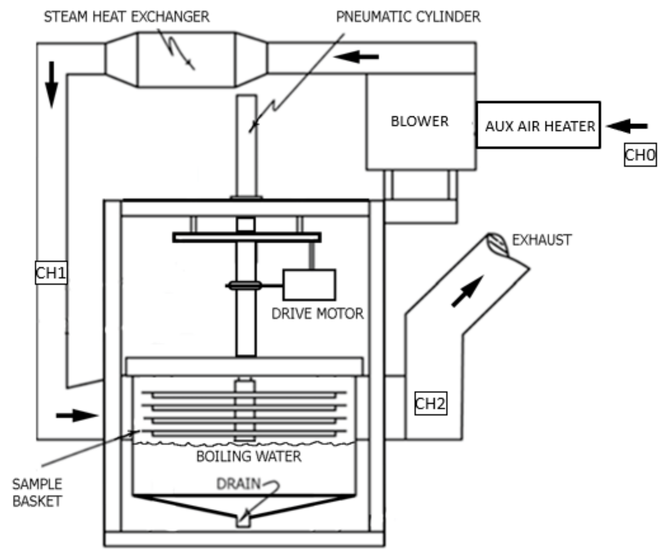

Figure 3-12, Thermocouple locations in the ABT (CH0, CH1 and CH2)

The ABT holds 24, pie-wedge, stainless steel wire baskets, which contain the specimens throughout all cycles as the carousel rotates at 3 rpm (Figure 3-13). The orientation of sample groups can be observed in Figure 3-13.

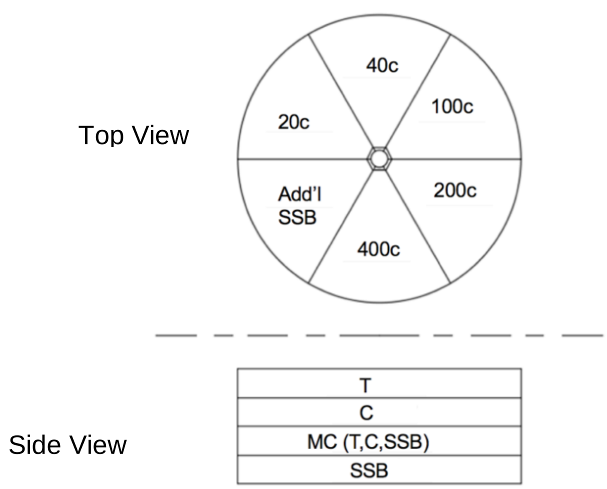

Figure 3-13, Sample basket arrangement inside the ABT, indicating placement of specimens for sampling after cycles 20, 40, 100, and 400.

Note that the SSB group required additional space due to the relatively large volume of the geometry. The MC specimen basket contains specimens for the segmented MC data described in section 3.2.3. As cycles were realized, specimens were removed from the sample baskets, then mechanically tested or segmented for MC determination. The emptied baskets were replaced inside the ABT and remaining specimens were reoriented so that maximum airflow and water circulation could be achieved

3.4 MCMEC

The MCMEC (multi-chamber modular environmental conditioning chamber) has three independently controlled environment chambers. There is forced-air circulation, with a lower temperature limit of -30°C. This system was employed in the BDF test in order to realize the harsh -30°C conditions the test requires for its respective freezing phase. A depiction of the MCMEC can be observed in Figure 3-14.

Figure 3-14, Multi-Chamber Modular Environmental Conditioning System

3.5 Vacuum Vessel with Band Heater

A vacuum vessel equipped with a band heater was used in order to maintain the 66 °C water temperature required by the VPS test. The volume of the vessel was ~26 liters, allowing enough space for each geometry group to be tested independently. A depiction of the vacuum vessel with the band heater can be observed in Figure 3-15.

Figure 3-15, Vacuum vessel with band heater

3.6 Wireless Transmitter

The temperature of the bondline was recorded using an Omega™ UWTC-2-NEMA wireless industrial transmitter. This transmitter has a tolerance of ± 1 °C, which can also be affected by electrical noise within the laboratory environment, in which the recording was conducted. The transmitter was attached to the specimen using a 30 AWG, type-K thermocouple. Temperature measurements were acquired in ten-second intervals and transmitted to a receiver linked to a local computer. A depiction of the wireless transmitter and thermocouple inserted into geometry C can be observed in Figure 3-16.

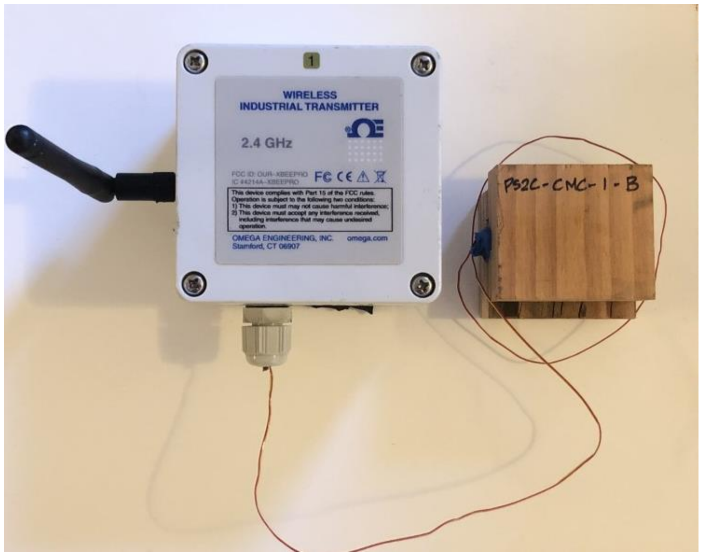

Figure 3-16, Compression Block Connected to Wireless Transmitter

3.7 Statistical Analysis

Statistical analysis for percent wood failure and mechanical results were conducted using Minitab 17 (2010). The maximum shear strength of all sample groups was recorded and compared by conducting a one-way ANOVA on all sample groups within one specimen geometry. This means that one geometry would be evaluated by comparing between all three weathering regimes including DC and WC conditions. Due to the large number of samples and possible comparisons, variability was assessed using boxplots and residual plots. Tukey pairwise comparison was conducted on individual geometries between cycles from the ABT including wet control, dry control, 20, 40, 100, 200, 400 and final cycles from the BDF and VPS test.

Strength retention of individual geometries was conducted by comparing all cycles from the ABT and the final cycles of the BDF and VPS test to the dry control sample group of their respective geometry. Outlier observations were removed on the basis that the data are at least 1.5 times the interquartile range (Q3 – Q1) from the edge of the box.

4. Results & Discussion

4.1 Mechanical Results

4.1.1 Lap-shear in Tension

Geometry T showed to have the highest variability amongst all treatments including DC, WC, cycles 20-400 of the ABT, and final cycles of the VPS and BDF tests. An increase in strength retention was observed in specimens from the 200 cycle group. This can be observed in Figure 4-1. This increase in strength retention is an artifact of the mechanical test and the natural variability of wooden substrates. Future studies should consider increasing the sample group from n=10 to n=30, allowing for a more robust analysis. A One-Way ANOVA with a 0.05 significance level was conducted on all sample groups, with the null hypothesis that all means are equal. A p-value of 0.00 was reported, indicating that the mean of at least one group is different than the others.

Tukey Pairwise comparisons indicate that the mean of the DC group is different than all other exposure conditions with the exception of the 20 cycle group. This indicates that significant loss in strength retention can be observed at cycle 40. Furthermore, specimens from the VPS and BDF sample groups do not differ in means, as indicated in Table 5. This indicated that the cyclic stresses from the BDF and VPS are similar for geometry T, regarding their impact on strength retention. Additionally, the mean of BDF did not differ from that of cycles 20-400 of the ABT, suggesting that these conditions are equivalent regarding their affect on strength retention. For the VPS group all ABT cycles with the exception of cycle 20 can be considered equivalent.

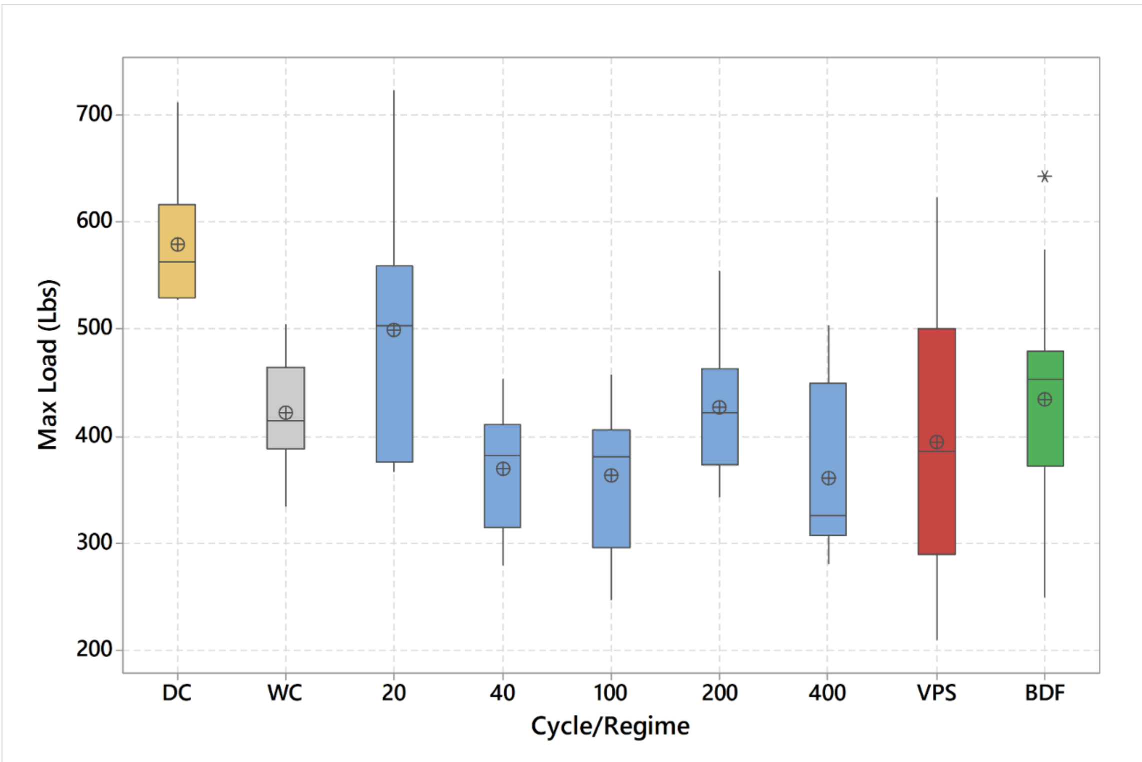

Figure 4-1, Mechanical results for geometry T: DC, WC, 400 Cycles ABT vs VPS vs BDF

Table 4-1, Analysis of variance for maximum load (lbf) of geometry T: 400 Cycles ABT, VPS, BDF

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Cycle	8	432942	54118	6.86	0.000
Error	120	946068	7884		
Total	128	1379010			

Table 4-2, Summary statistics from one-way ANOVA, with 95% confidence intervals for maximum load (lb_f) of geometry T: DC, WC, 400 Cycles ABT, VPS, BDF

Cycle	N	Mean	StDev	95% CI
DC	10	578.9	61.0	(523.3, 634.5)
WC	10	421.4	52.5	(365.8, 477.0)
20	10	498.3	114.0	(442.8, 553.9)
40	10	370.0	57.4	(314.5, 425.6)
100	11	363.6	64.7	(310.6, 416.6)
200	10	426.3	66.1	(370.7, 481.9)
400	10	361.0	80.5	(305.4, 416.6)
VPS	29	394.0	118.1	(361.4, 426.7)
BDF	29	433.9	86.8	(401.2, 466.5)

Table 4-3, Tukey pairwise comparisons for maximum load (lb_f) of geometry T (confidence coefficient = 0.95): DC, WC, 400 Cycles ABT, VPS, BDF

Cycle	N	Mean	Grouping
DC	10	578.9	A
WC	10	421.4	B C
20	10	498.3	A B
40	10	370.0	C
100	11	363.6	C
200	10	426.3	B C
400	10	361.0	C
VPS	29	394.0	C
BDF	29	433.9	B C

4.1.2 Lap-shear in Compression w/ PF

Geometry C followed a similar trend to that of T, but realized the greatest loss in strength retention amongst all three weathering regimes. While cycle 400, VPS and BDF proved to be the most arduous for this geometry, their individual means did not differ from one another. This is indicated by their overlapping confidence intervals (Table 4-5). The results of this test could be accounted by considering the relative volume of wood of geometry C to that of the geometry T, which are 5.25 in^3 and 0.81 in^3, respectively. Although both employed the same adhesive and spread rate, the relative volume of wood could imply that the larger substrate was more responsible for loss in strength retention than that of geometry T.

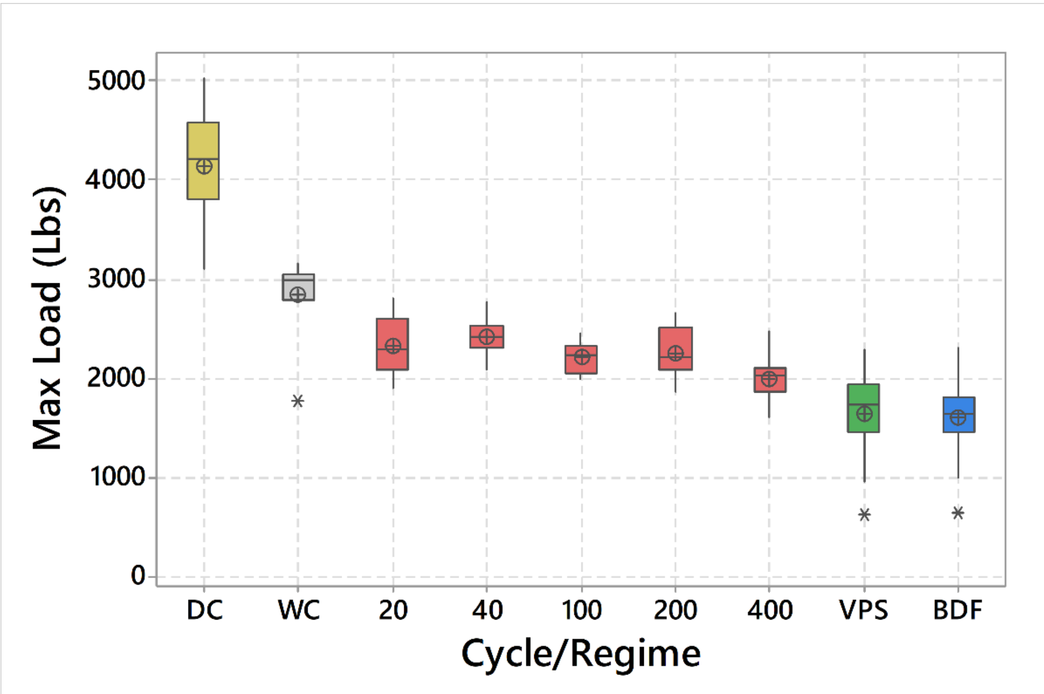

Figure 4-2, Mechanical results for C w/ PF geometry: DC, WC, 400 Cycles ABT vs VPS vs BDF

Table 4-4, Analysis of variance for maximum load (lb$_f$) of geometry C: DC, WC, 400 Cycles ABT, VPS, BDF

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Cycle	8	61958123	7744765	64.96	0.000
Error	119	14187033	119219		
Total	127	76145156			

Table 4-5, Summary statistics from one-way ANOVA, with 95% confidence intervals for maximum load (lb_f) of geometry C: DC, WC, 400 Cycles ABT, VPS, BDF

Cycle	N	Mean	StDev	95% CI
DC	10	4138.0	593.0	(3921, 4354)
WC	10	2837.0	400.0	(2621, 3054)
20	10	2324.3	296.9	(2108, 2540)
40	10	2414.0	194.3	(2198, 2630)
100	11	2206.0	148.6	(2000, 2412)
200	10	2252.2	253.2	(2036, 2468)
400	9	1993.5	239.8	(1766, 2221)
VPS	29	1637.7	378.6	(1511, 1765)
BDF	29	1600.5	336.8	(1474, 1728)

Table 4-6, Tukey pairwise comparisons for geometry C (confidence coefficient = 0.95): 400 Cycles ABT, VPS, BDF

Cycle	N	Mean	Grouping
DC	10	4138.0	A
WC	10	2837.0	B
20	10	2414.0	B C
40	10	2324.3	C
100	10	2252.2	C
200	10	2206.0	C
400	10	1993.5	C D
VPS	29	1637.7	D
BDF	29	1600.5	D

4.1.3 Lap-shear in Compression w/ PU

CPU was only exposed and evaluated within the ABT regime. The trend showed to be consistent with that of C w/ PF. Significant loss in strength was observed after the WC exposure. Within the active ABT cycle, significant lost in strength retention was observed at cycle 20. There was no observed difference in means amongst cycles 20, 40, 100, 200 and 400.

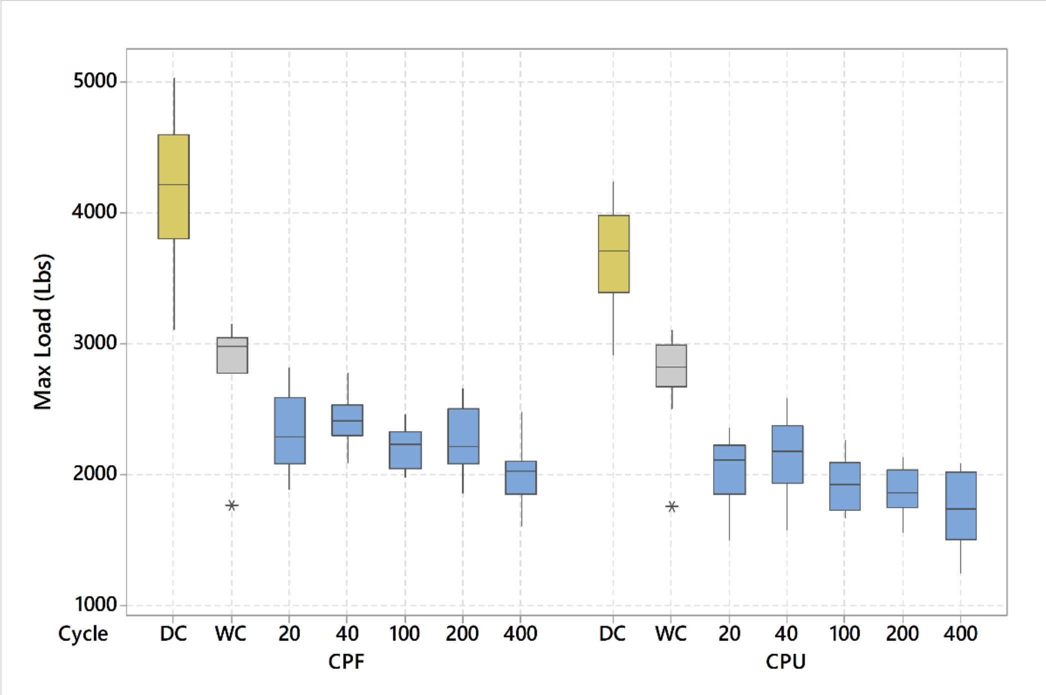

Figure 4-3, Mechanical results for CPU geometry, 400 Cycles ABT

Table 4-7, Analysis of variance for maximum load (lb$_f$) of geometry CPU: 400 Cycles ABT

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Cycle	6	27304877	4550813	49.55	0.000
Error	63	5785645	91836		
Total	69	33090521			

Table 4-8, Summary statistics from one-way ANOVA, with 95% Confidence intervals for maximum load (lb$_f$) of geometry CPU: DC, WC, 400 Cycles ABT

Cycle	N	Mean	StDev	95% CI
DC	10	3654.0	404.0	(3463, 3846)
WC	10	2745.0	389.0	(2553, 2936)
20	10	2026.3	289.0	(1835, 2218)
40	10	2148.9	298.2	(1957, 2341)
100	11	1946.9	201.9	(1764, 2130)
200	10	1869.6	183.4	(1678, 2061)
400	9	1743.4	294.3	(1542, 1945)

Table 4-9, Tukey pairwise comparisons for maximum load (lb_f) of geometry CPU (confidence coefficient = 0.95): 400 Cycles ABT

Cycle	N	Mean	Grouping
DC	10	3654.0	A
WC	10	2745.0	B
20	10	2148.9	C
40	10	2026.3	C
100	11	1946.9	C
200	10	1869.6	C
400	9	1743.4	C

4.1.4 Short-span Shear in Bending

The SSB group followed a similar trend to the C group, displaying a clear decreasing trend in strength retention. Within cycles from the ABT, only cycles 100, 200 and 400 showed to be more severe than that of the WC treatment. There was no significant difference in means of VPS and cycle 400 groups. While the VPS and BDF groups did not differ in means, the greatest loss in strength retention was observed in the VPS group.

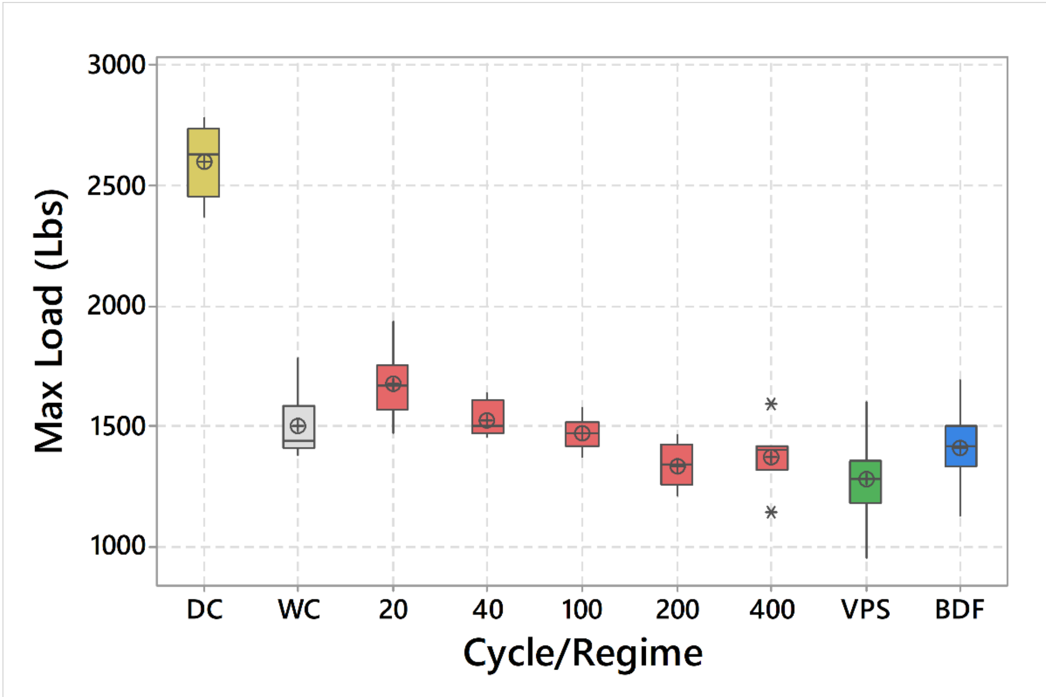

Figure 4-4, Mechanical results for SSB geometry, 400 Cycles ABT vs VPS vs BDF

Table 4-10, Analysis of variance for maximum load (lb_f) of geometry SSB: 400 Cycles ABT, VPS, BDF

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Cycle	8	14593571	1824196	110.49	0.000
Error	119	1964652	16510		
Total	127	16558224			

Table 4-11, Summary statistics from one-way ANOVA, with 95% confidence intervals for maximum load (lb_f) of geometry SSB: DC, WC, 400 Cycles ABT, VPS, BDF

Cycle	N	Mean	StDev	95% CI
DC	10	2601.8	148.1	(2521, 2682)
WC	10	1496.8	133.9	(1416, 1577)
20	10	1676.1	144.3	(1596, 1757)
40	10	1525.6	69.1	(1445, 1606)
100	11	1467.0	65.7	(1390, 1544)
200	10	1334.0	93.7	(1254, 1414)
400	10	1370.3	119.8	(1286, 1455)
VPS	29	1274.5	145.5	(1227, 1322)
BDF	29	1411.3	139.6	(1364, 1459)

Table 4-12, Tukey pairwise comparisons for maximum load (lb_f) of geometry SSB: 400 Cycles ABT, VPS, BDF

Cycle	N	Mean	Grouping
DC	10	2601.8	A
WC	10	1676.1	B
20	10	1525.6	B C
40	10	1496.8	B C D
100	11	1467.0	C D
200	29	1411.3	C D
400	10	1370.3	C D E
VPS	10	1334.0	D E
BDF	29	1274.5	E

4.1.5 Non-linear Regression Analysis

Data from all three specimen geometries for cycles 20-400 was compared to the mean of their respective DC group. VPS and BDF groups were plotted by fitting their mean percent strength retention values against that of the DC group from their respective geometry. WC groups were not included because they were not exposed to active ABT cycles, but rather a 72 hr. water soak. The fitted non-linear regression equations for each geometry are listed in Table 4-13.

The residual plots revealed the model was unbiased. The model explained 42%, 72% and 86% of the variation of percent strength retention for the lap-shear in tension loading, compression shear block, and short-span bending results, respectively, as a function of cyclic exposure in the automatic boil test procedure.

Table 4-13, Non-linear regression equations for geometries T, C and SSB: DC, 400 Cycles ABT

Geometry	Equation	R^2
T	y = -0.063ln(x) + 0.9828	0.417
C	y = -0.082ln(x) + 0.9279	0.725
SSB	y = -0.082ln(x) + 0.9474	0.864

With a known percent strength retention, the VPS and BDF test can be compared directly to ABT cycles, within one geometry. Equivalent strength retention of geometry T for the VPS and BDF tests were realized at ABT cycles 120 and 35, respectively. For the SSB geometry, equivalent strength retention was found to be at the ABT's cycle 125 for the BDF test and 250 for the VPS test. As observed in Figure 4-6, geometry C fell beyond cycle 400, but not outside of the specifications of ASTM D3434's 1600 cycles. For the VPS and BDF test, strength retention for C achieved a predicted fit at cycle 650 and 770 of the ABT based on extrapolation of the fitted model.

Table 4-14, Equivalent % strength retention for geometries T, C and SSB: 400 cycles ABT vs BDF vs VPS

Geometry	*Test*	*Cycle ABT*	*%* SR
T	VPS	120	69%
T	BDF	35	76%
C	VPS	650	39%
C	BDF	770	38%
SSB	VPS	250	49%
SSB	BDF	125	54%

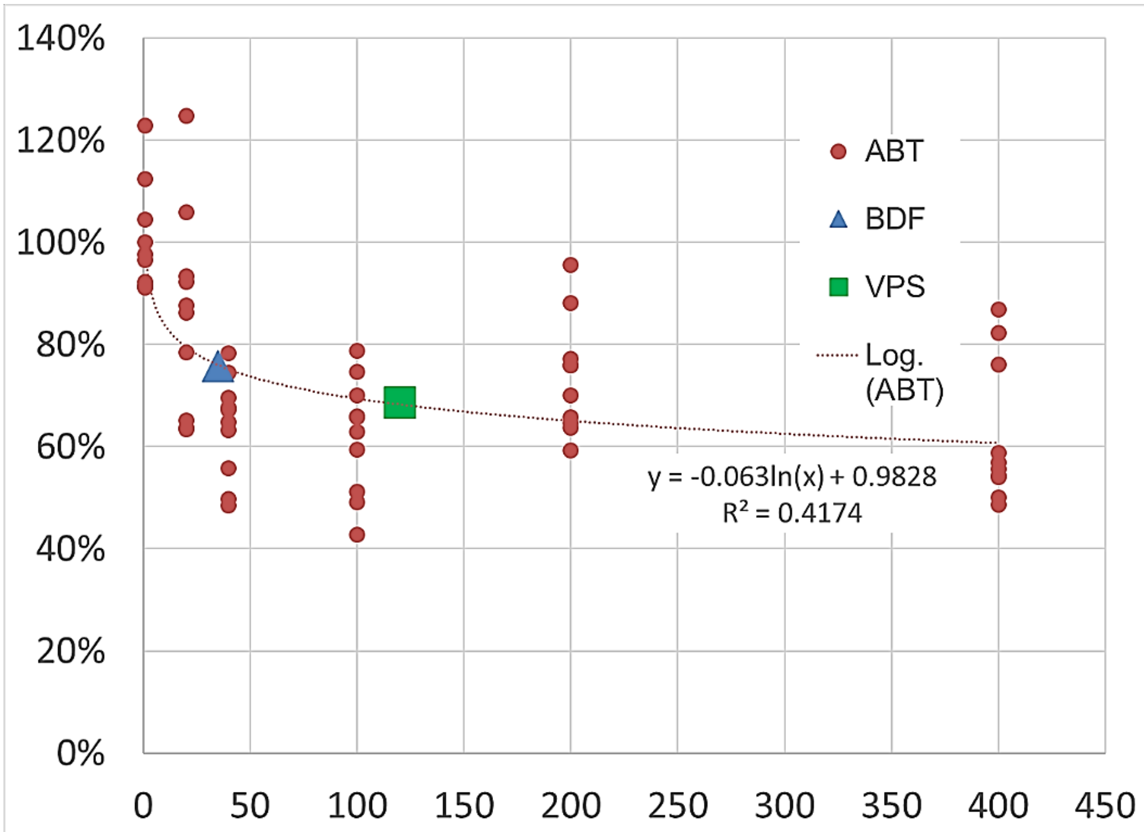

Figure 4-5, Non-linear regression for percent strength retention of geometry T, 400 Cycles ABT vs VPS vs BDF

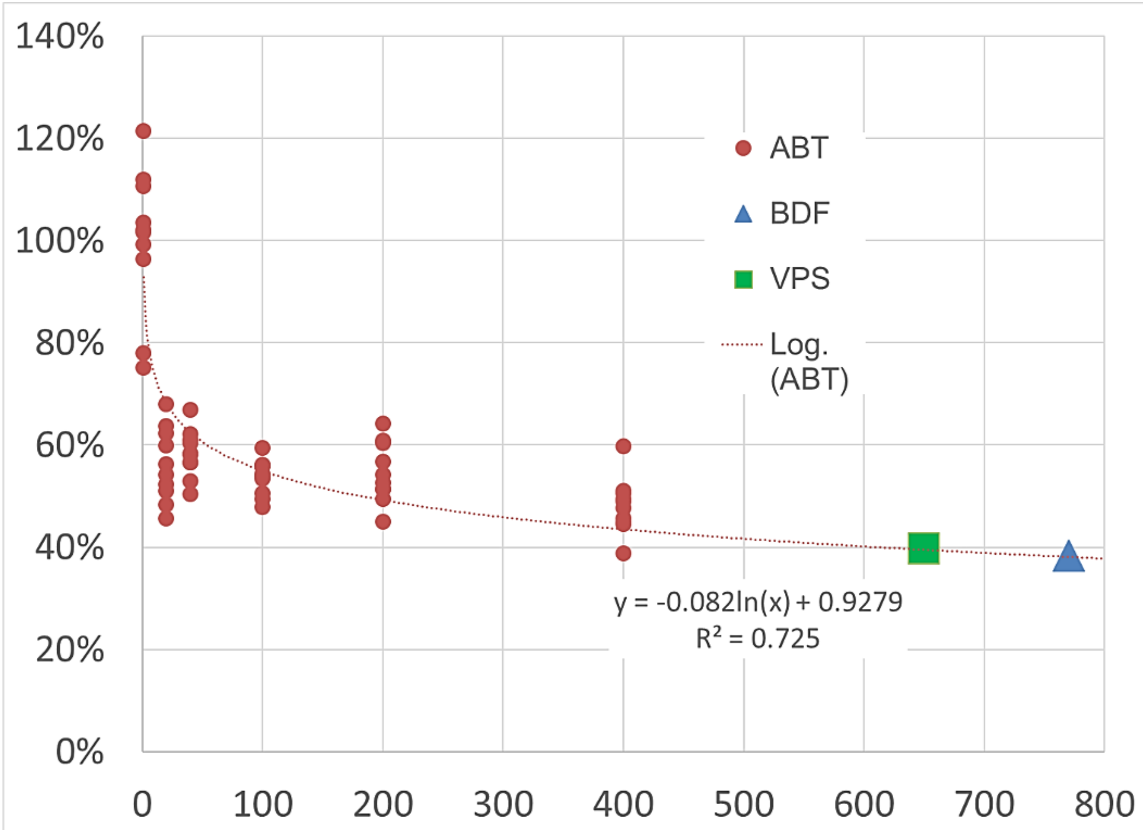

Figure 4-6, Non-linear regression for percent strength retention of geometry C, 400 Cycles ABT vs VPS vs BDF

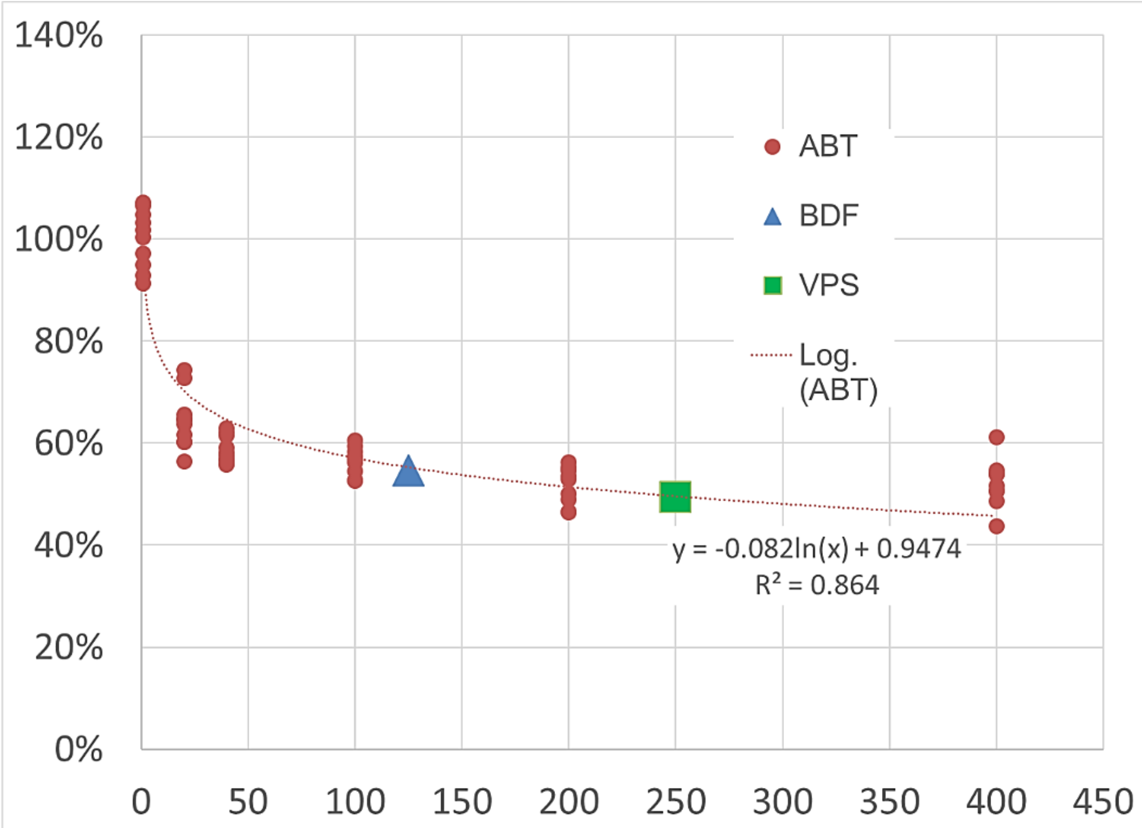

Figure 4-7, Non-linear regression for percent strength retention of geometry SSB, 400 Cycles ABT vs VPS vs BDF

4.2 Moisture Content

4.2.1 ABT

The ABT subjects specimens to cyclic conditions in an effort to achieve boiling and drying, which effectively strains the adhesive bondline. All specimen geometries increased moisture content throughout 400 cycles of exposure. Geometry C deviated from its upward trend at cycle 200 (Figure 4-9). Specimens from this group were most likely contained in a location of the ABT that allowed greater access to airflow during the dry phase between cycles 100 and 200. While geometries C and T quickly reached boiling point, neither geometry was given sufficient opportunity to dry below FSP, throughout all 400 cycles. All three geometries continued to ingress moisture without cyclic drying, reducing desired stress on the bondline. This can be observed in Figure 4-8 through Figure 4-10.

Again, this would imply that the ABT’s dry phase is not long enough. Future studies should consider increasing the length of the dry phase substantially. Alternatively, specimens could be separated using spacers, as Wilke (1977) suggested, allowing maximum air circulation around each specimen. ASTM D3434 neglects to specify this in the standard.

It should be noted that the ABT was calibrated to meet the requirements of ASTM D3434 for air velocity and temperature. However, these measurements were made in the air duct adjacent to the chamber. Inside the chamber, and above the boiling water, the air velocity is reduced due to the large cross-section area of the chamber in comparison to the cross-section of the inlet air duct. In addition, evaporation of the boiling water in the chamber would cool the inlet air, which was measured at roughly 90°C, instead of 107°C that was measured inside the inlet air duct. Reduced air velocity and reduced temperature are conditions that would lower drying rate during the drying phase of the ABT cycle.

The ABT subjects the specimens to dynamic conditions that cause adsorption and desorption of water depending on the phase of the cycle. The bondline position (center of specimen) would be the slowest to gain water during boiling, and the slowest to lose water during drying. The time allowed for boiling and drying was drastically different, at 10 min and 57 min, respectively. Clearly, the water adsorption phase dominated the water desorption phase. After 100 cycles, a maximum MC of approximately 190%

appears to have been achieved in the T geometry, which were the smallest specimens. The largest specimens, with SBB geometry, were below 140% MC after 400 cycles.

In the lap-shear specimens in tension loading, the bondline MC was consistently lower than the surface layers after 100 cycles. With the compression shear block specimens there was no consistent trend of MC of the bondline compared to the surface layers. The MC of the primary bondline in the short-span shear specimens was consistently greater than the MC of the surface layers. This indicates that some drying of the surface layers does occur in the SBB specimens. The differences of MC observed between the three specimen geometries were surely caused by the difference in thickness, volume, and relative surface area of the transverse plane.

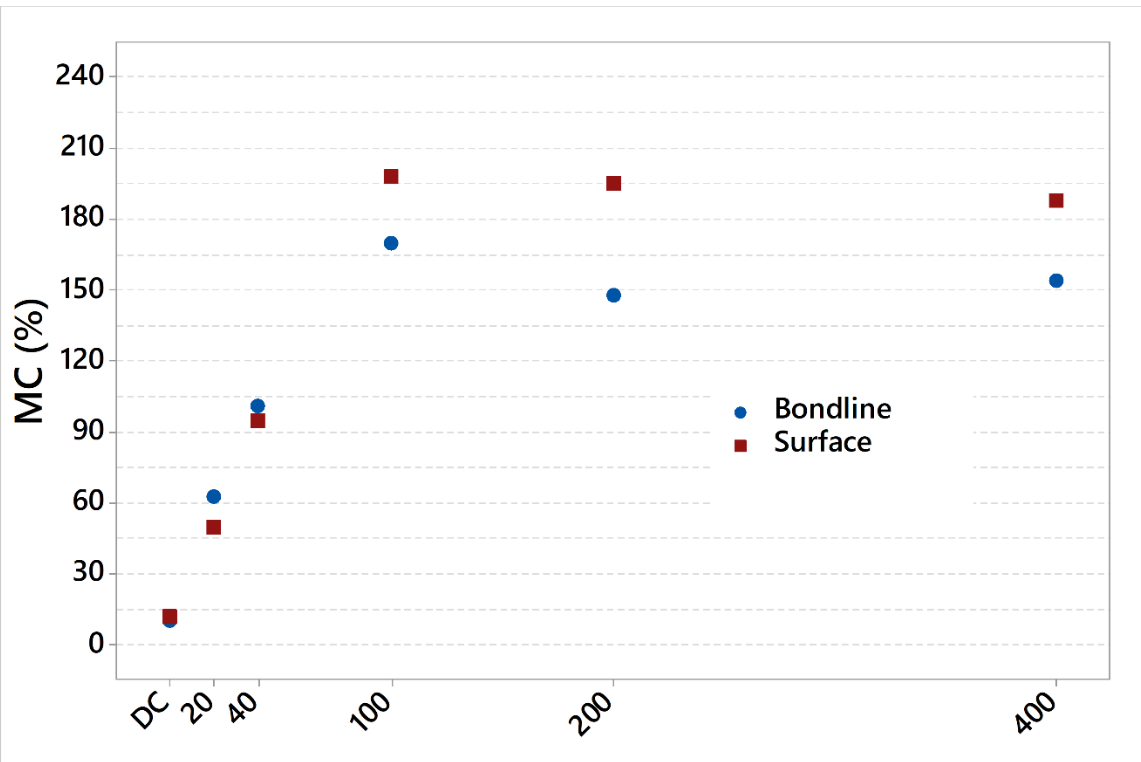

Figure 4-8, MC results after drying phase for geometry T, 400 Cycles ABT

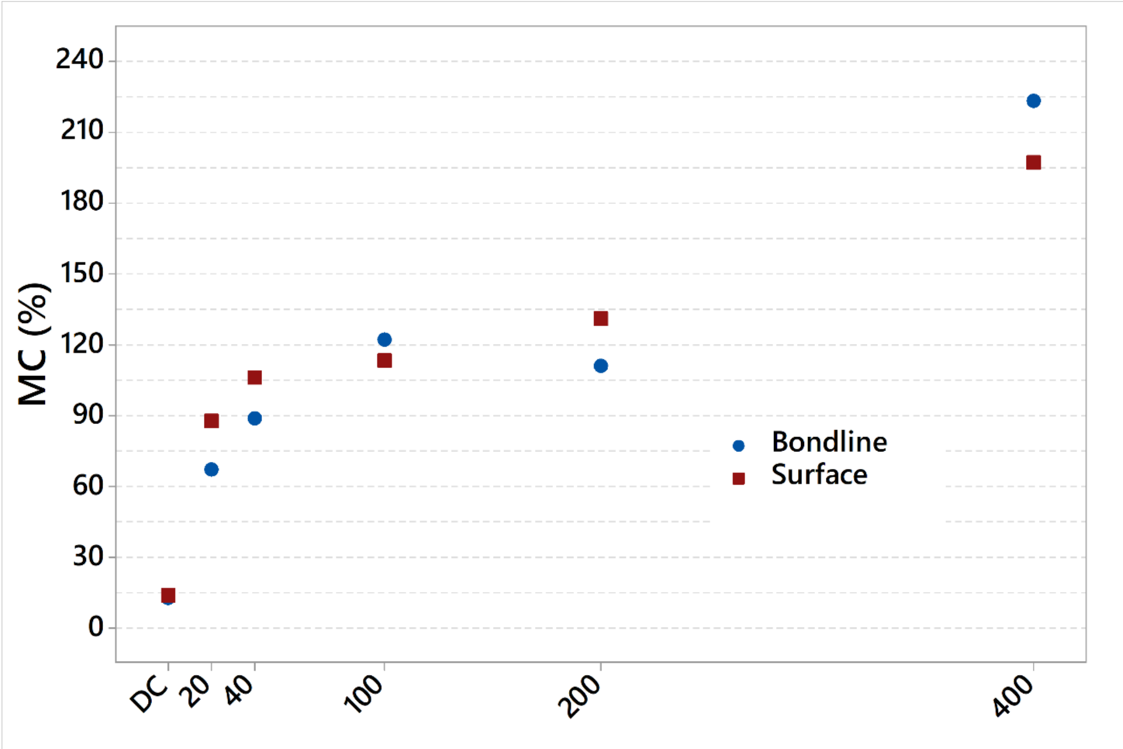

Figure 4-9, MC results after drying phase for geometry CPF, 400 Cycles ABT

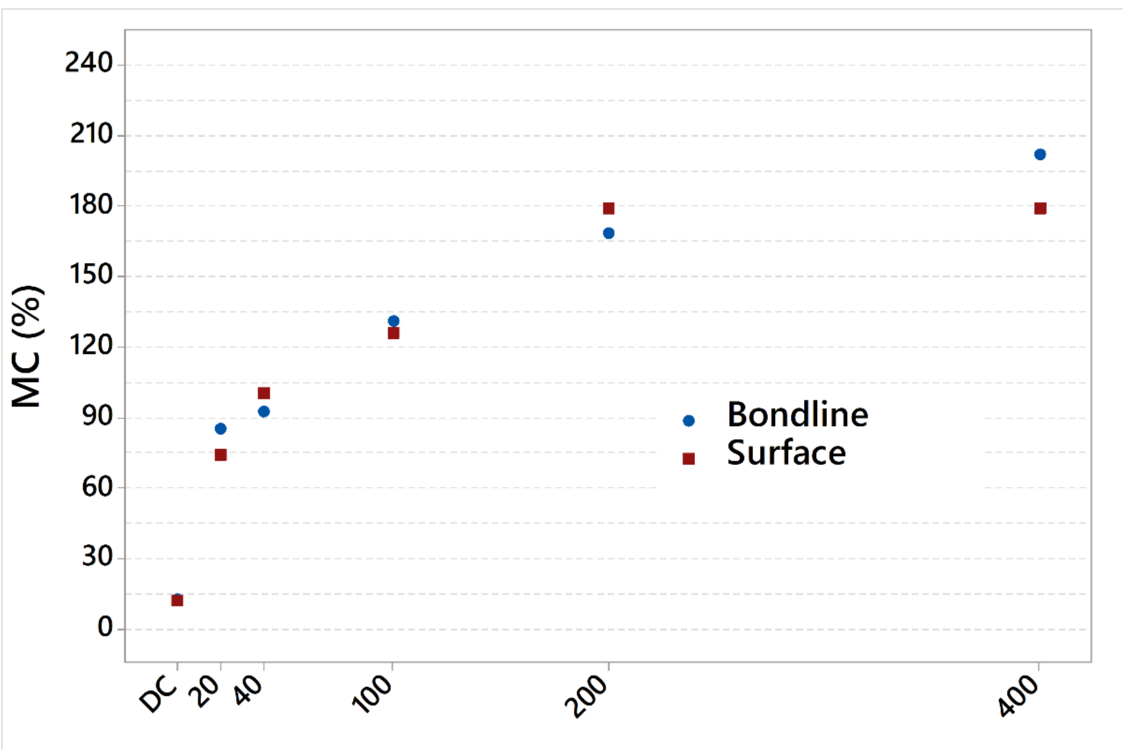

Figure 4-10, MC results after drying phase for geometry CPU, 400 Cycles ABT

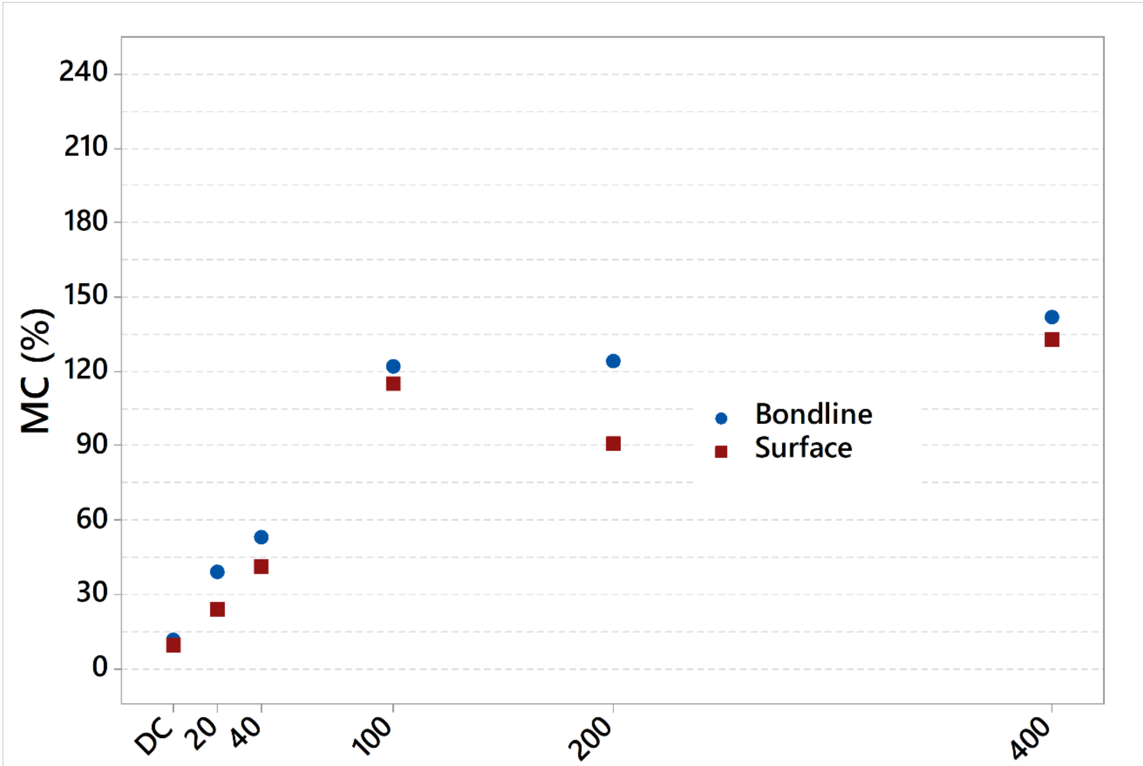

Figure 4-11, MC results after drying phase for geometry SSB, 400 Cycles ABT

4.2.2 VPS

Unlike the ABT, the VPS test proved to dry specimens effectively. The fluctuating trend observed is most accurately explained by the alternating 6hr and 15hr dry phases specified by PS2 7.17. While specimens clearly dried more during the 15hr period, both 6hr and 15hr phases drove the MC of all geometries below FSP. The bondline MC of geometry C and T was consistently higher than the surface measurements, for all but cycle 2 for geometry T and cycle 1 for geometry C. This suggests that the oven allowed the surface to dry at a higher rate than the bondline for both geometries. The SSB geometry showed to have a fluctuating trend between surface and bondline measurements. This is likely explained by the relatively larger volume of the SSB geometry.

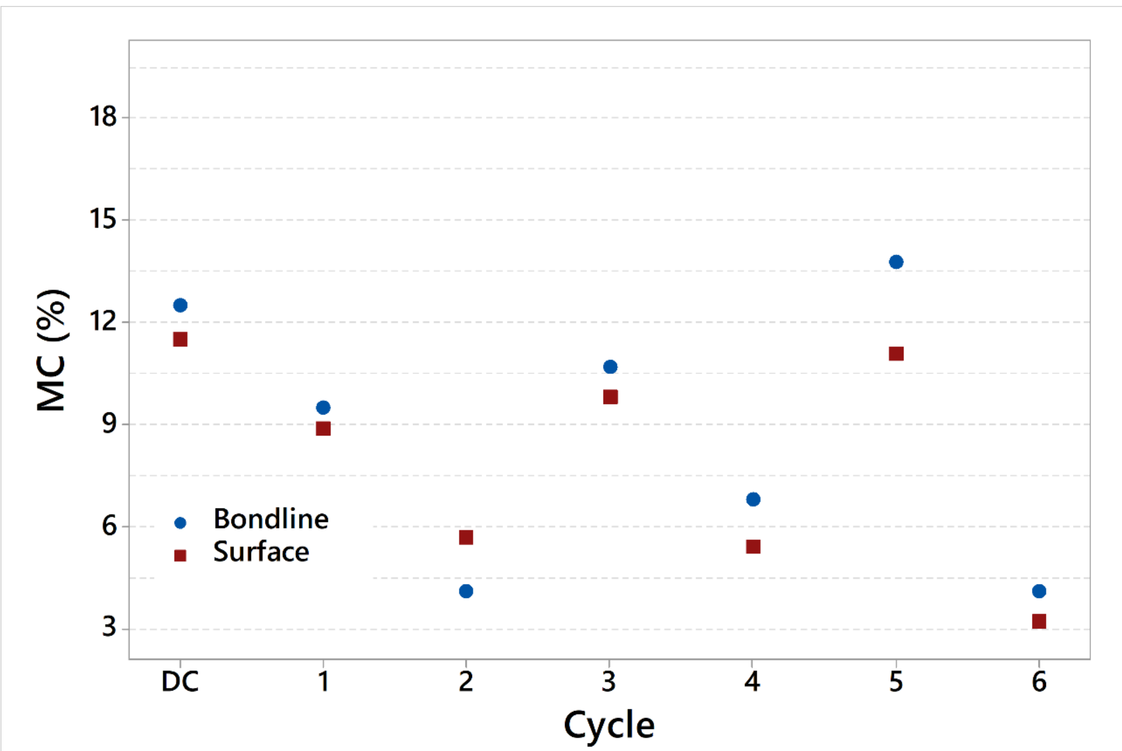

Figure 4-12, MC results after drying phase for geometry T, VPS

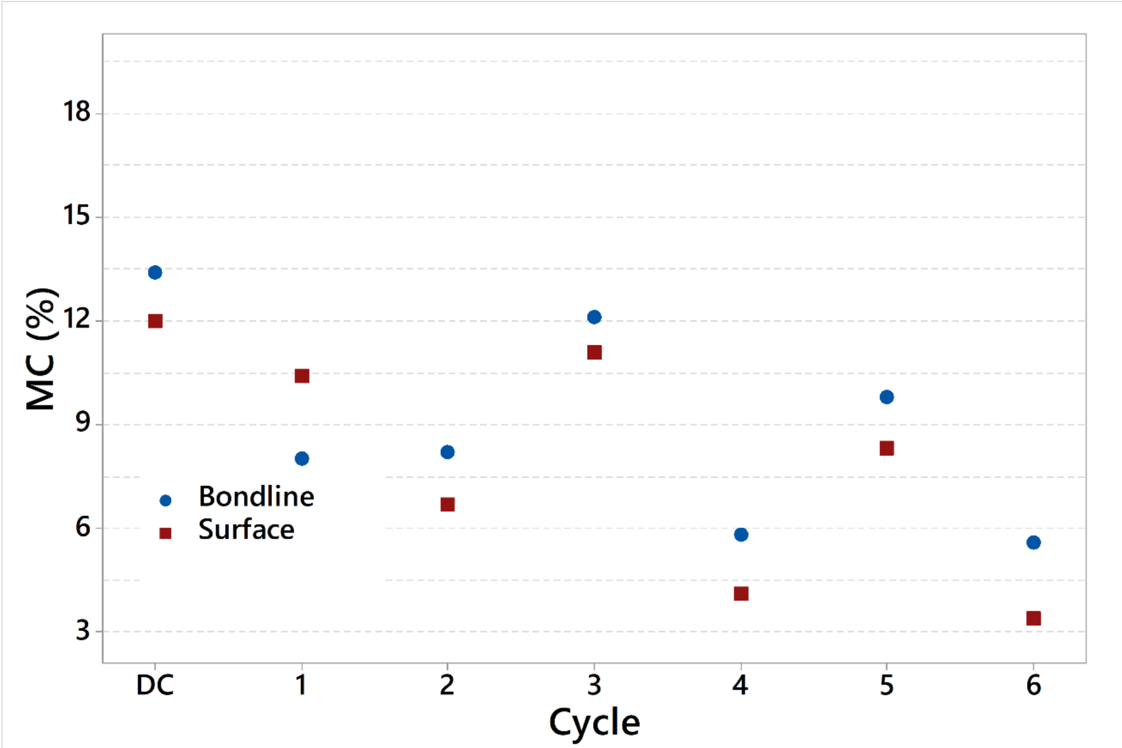

Figure 4-13, MC results after drying phase for Geometry SSB, VPS

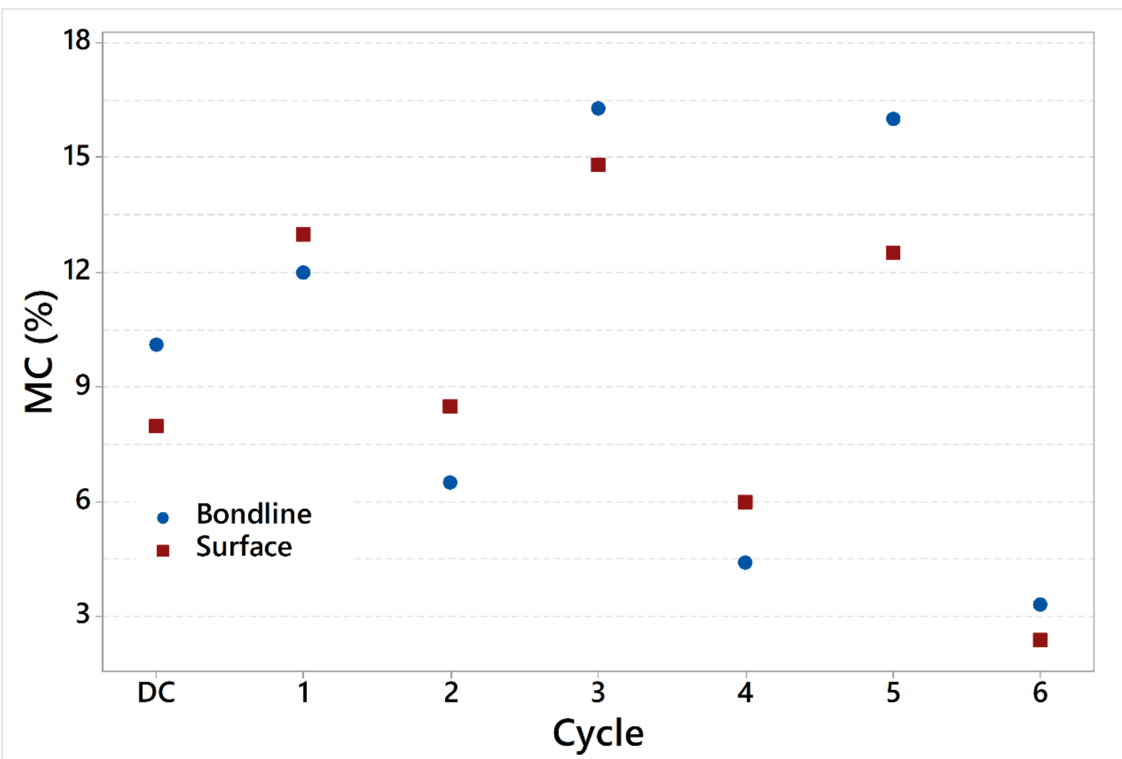

Figure 4-14, MC results after drying phase for Geometry C, VPS

4.2.3 BDF

Similar to the VPS test, specimen geometries subjected to the BDF test showed to bring all three geometries below FSP, suggesting that the 19hr dry phase is sufficient. Geometry T had showed the highest variability in MC following the dry phase of the BDF test. Unlike the VPS test, all geometries' surface and bondline were dried consistently, displaying a downward trend as the cycles advanced. The SSB geometry showed the least variability between bondline and surface measurements. Again, this is due to the homogeneity of LVL.

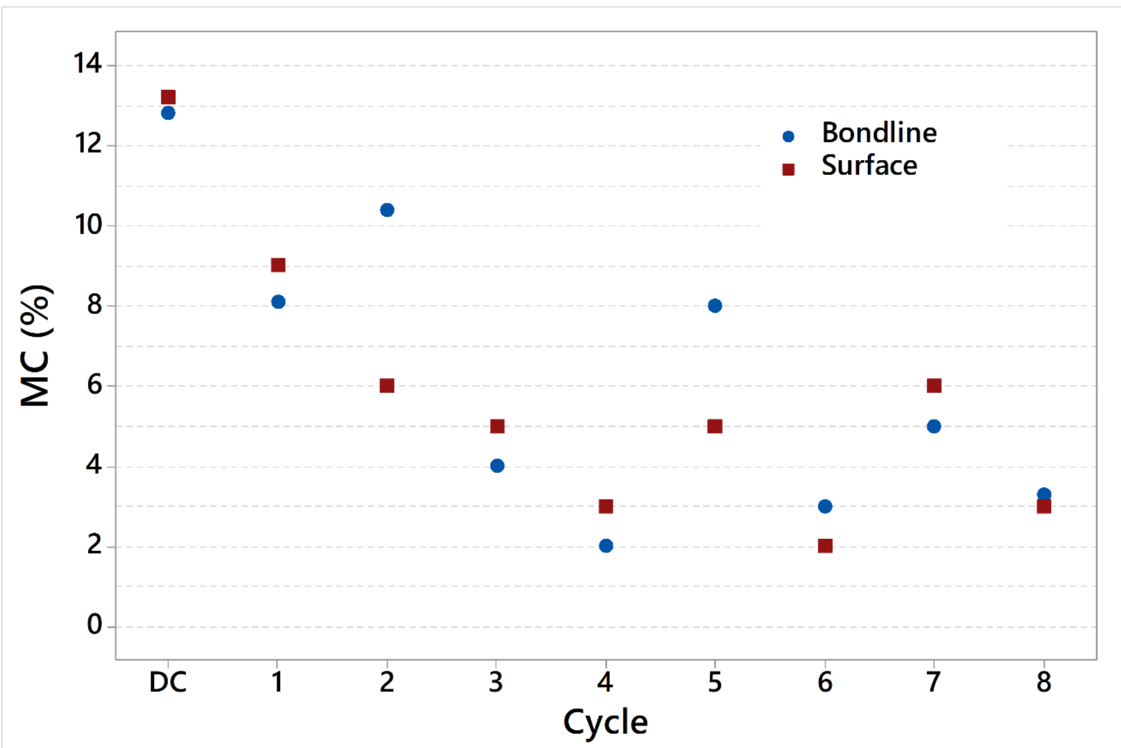

Figure 4-15, MC results after drying phase for Geometry T, BDF

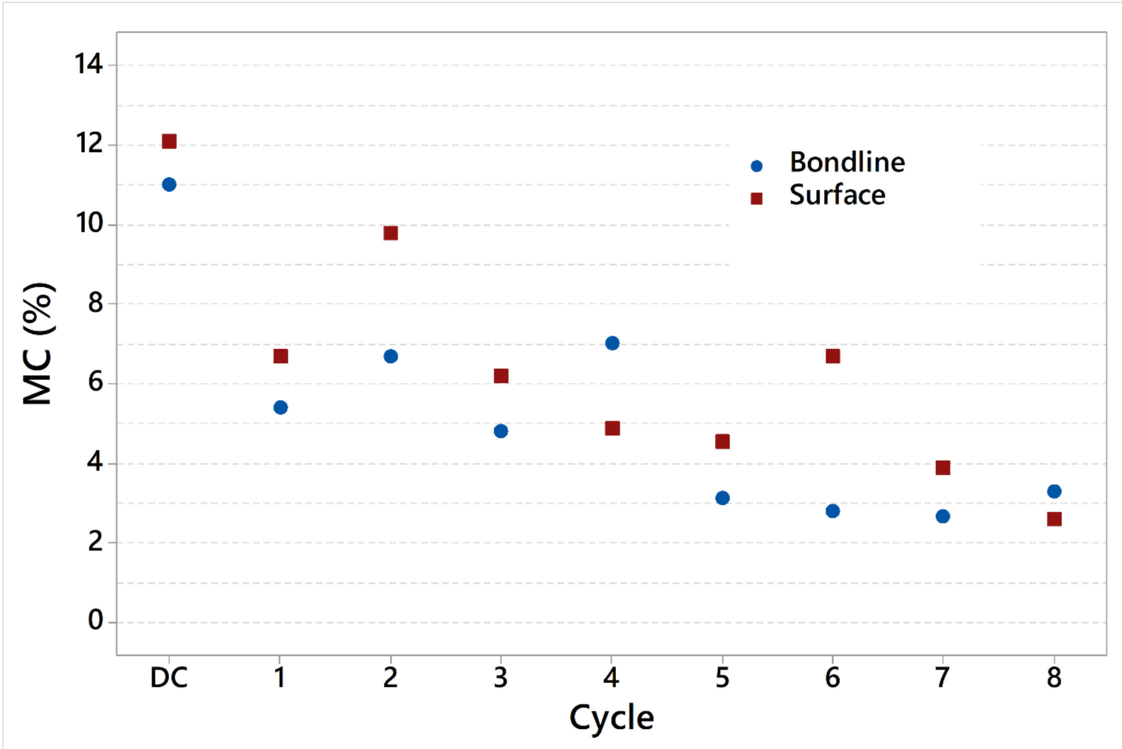

Figure 4-16, MC results after drying phase for Geometry C, BDF

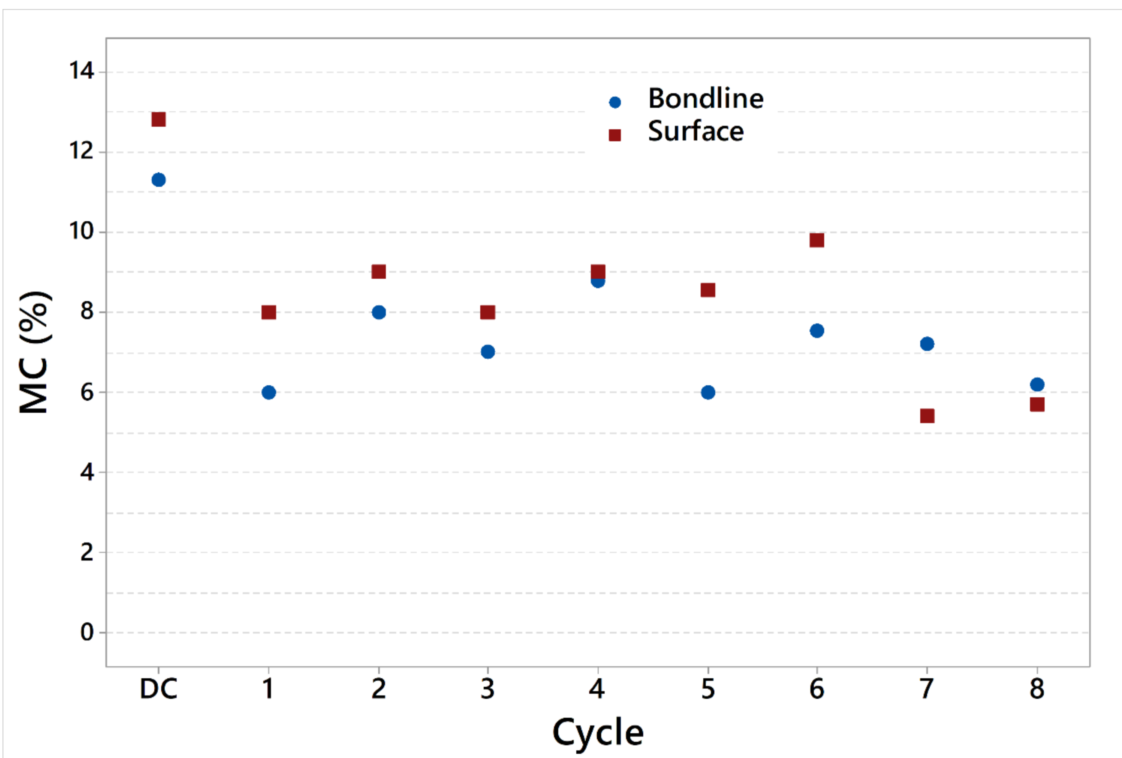

Figure 4-17, MC results after drying phase for Geometry SSB, BDF

4.3 Bondline Temperature Result

4.3.1 ASTM D3434 – ABT

A recapitulation of exposure conditions for the Automatic Boil Test are displayed in Table 4-15.

Table 4-15, Exposure Conditions for The Automatic Boil Test

ASTM D3434 – Automatic Boil Test
1. Boil 10 min
2. Cool at 23°C, 4 min
3. Dry at 107°C, 57 min
4. Repeat steps 1-3 for 400 cycles

Bondline temperature conditions for each geometry exposed to the ABT can be observed in *Figure 4-18* through Figure 4-20. Although RH was approximated to be 10%, bondline temperature results for all three geometries showed that the dry phase is clearly not long enough. While the bondline of geometries C and T clearly approached or achieved boiling point within the 10 min exposure of the phase, neither of them exceeded 75°C during the 107°C and 57 min dry phase. The dry phase for geometry T appears to be longer than 57 minutes. This is due to the geometries ability to cool quickly during the cooling phase, ultimately making the dry phase appear to be longer than that of C and SSB.

With an excess of free water present in the specimens, one expects the specimen surface temperature to reach 100°C, if the air temperature exceeded 100°C. Therefore, the air temperature inside the chamber, once mixed with evaporating water, was less than 100°C. This demonstrates that the dry phase was not extensive enough to drop the MC below the FSP. This trend can be observed in Figures 4-8 through 4-11. There was no observed change in the temperature trend after 3 cycles for all geometries.

The SSB geometry also faced similar temperature results during the dry phase, with the bondline temperature never exceeding 65°C, which was the lowest temperature among the three specimen geometries. The relatively large size of the SSB geometry prevented the bondline from reaching the boiling temperature throughout all 400 cycles of exposure. The SBB specimens experienced the lowest extremes of temperature at the bondline.

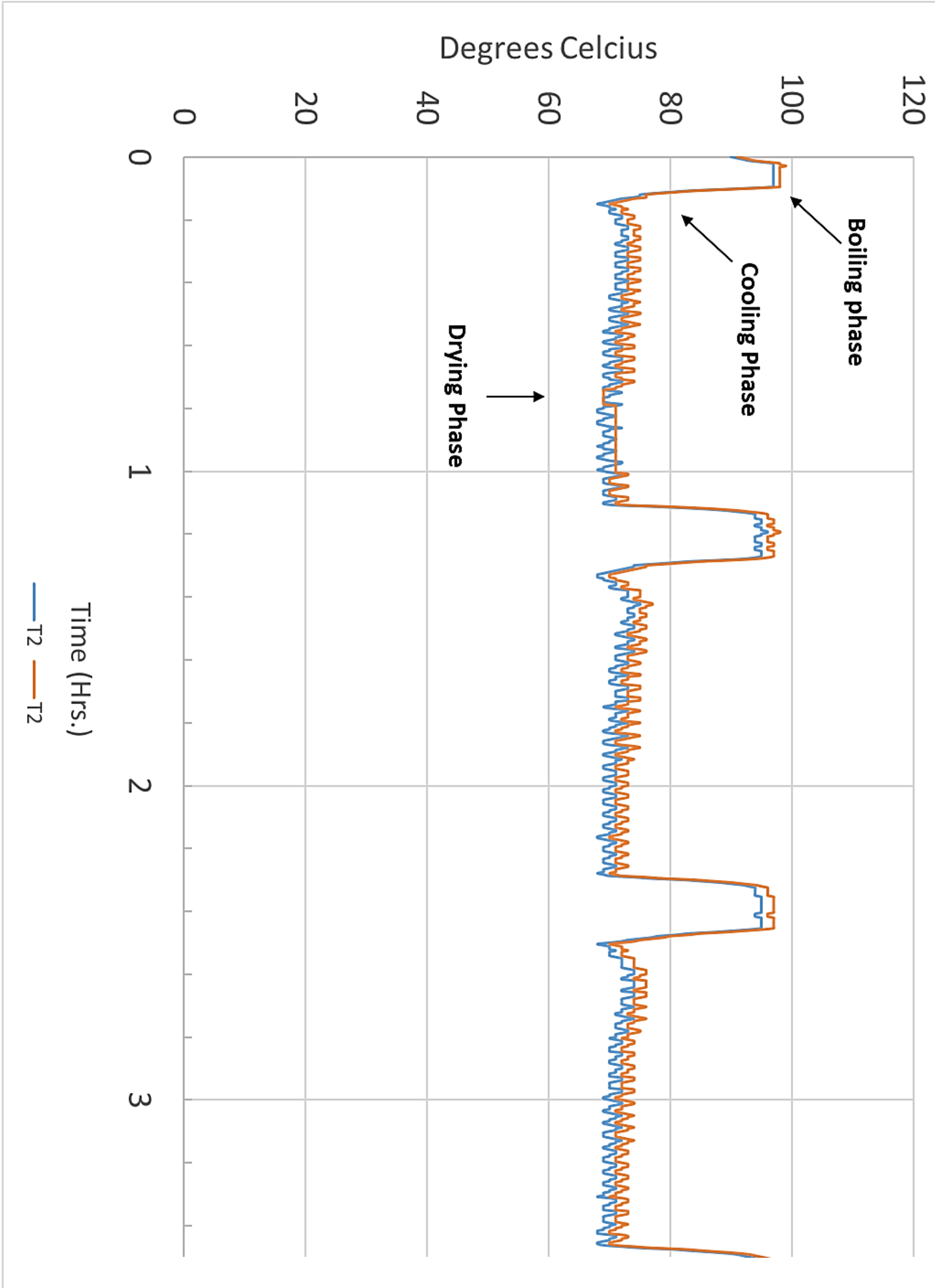

Figure 4-18, Bondline temperature results for Geometry T, 3 cycles ABT

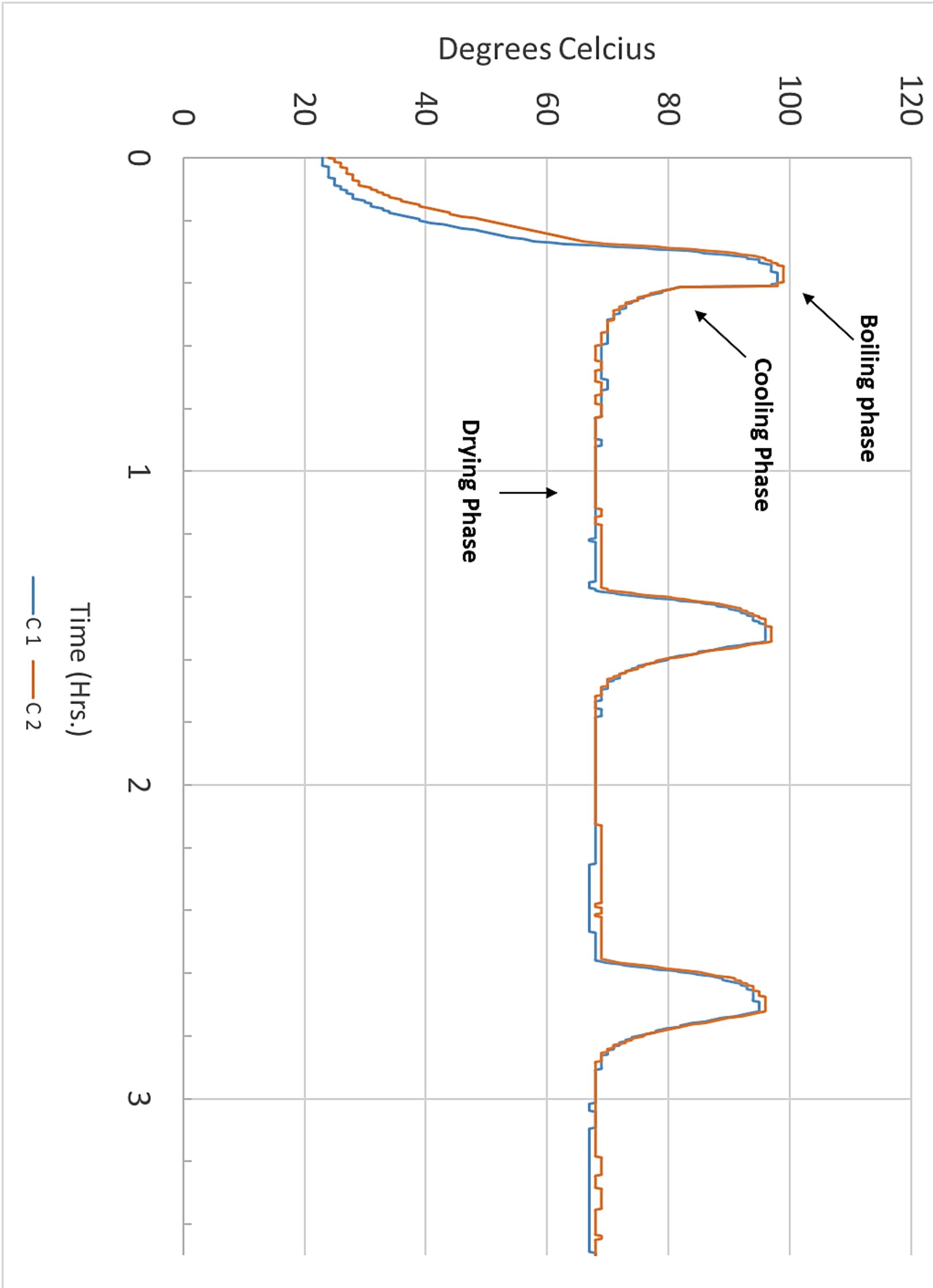

Figure 4-19, Bondline temperature results for Geometry C, 3 cycles ABT

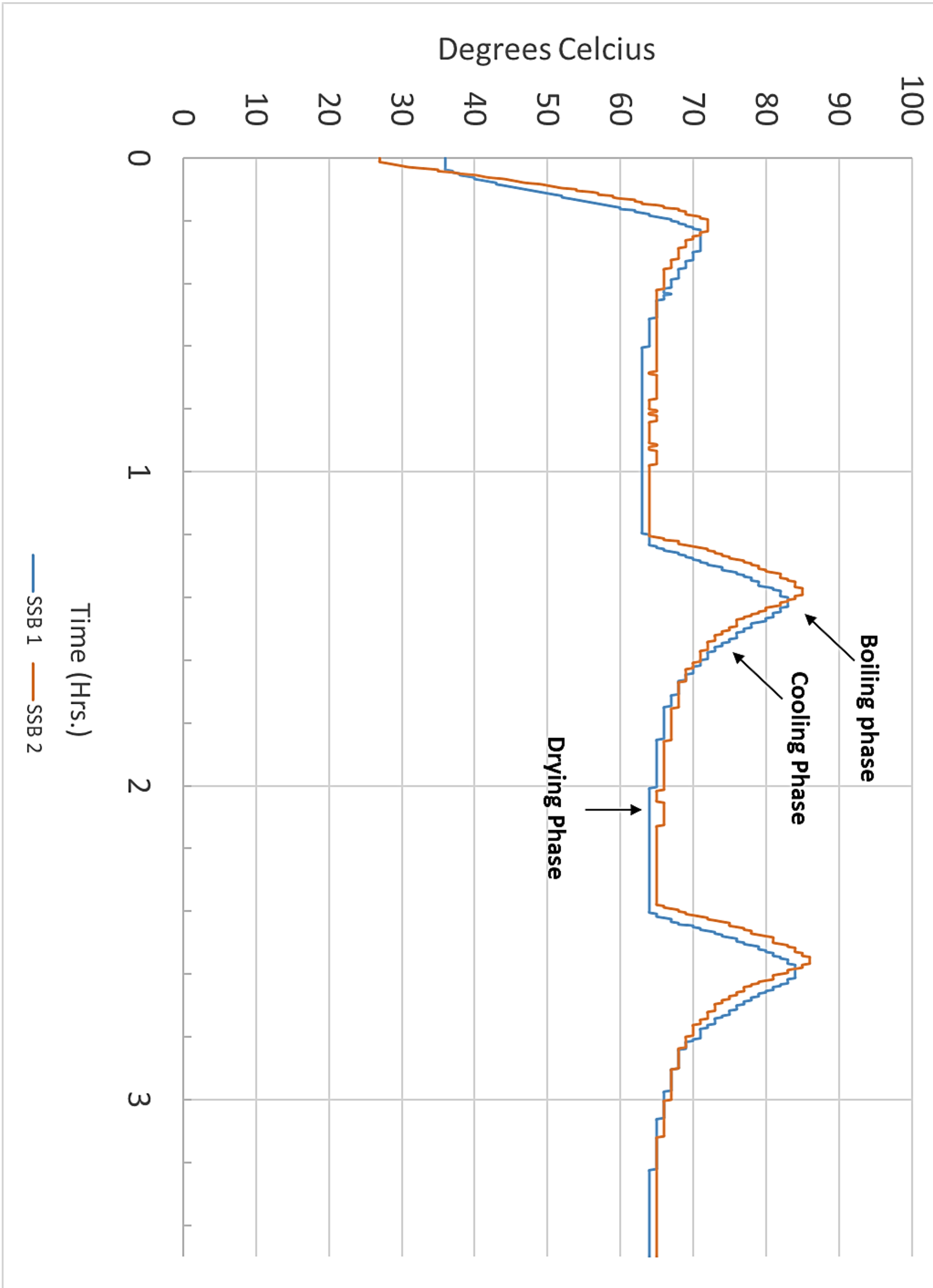

Figure 4-20, Bondline temperature results for Geometry SSB, 3 cycles ABT

4.3.2 PS2 7.17 – VPS

A recapitulation of exposure conditions for the VPS test is represented in Table 4-16.

Table 4-16, Exposure Conditions for 6-Cyle Vacuum Pressure Soak

PS2 7.17– 6-Cycle Vacuum Pressure Soak
1. 30 min in 66 °C water with a vacuum of 50.6 kPa (15 in. Hg)
2. 30 min in 66 °C water at ambient pressure
3. Dry at 82°C for 6hrs.
4. Repeat steps 1 and 2, then dry at 82°C for 15hrs.
5. This completes two cycles
6. Repeat 4 more cycles

The bondline temperature results for the VPS test showed that all geometries reached the temperature of the exposure conditions throughout cyclic exposure of the VPS. There are some differences between the specimen geometries to note. The smallest specimen, geometry T, had the fastest response to temperature change at the bondline, with approximately 190 min required to reach the specified oven temperature. The largest specimen, geometry SBB, required about 8 hours to reach oven temperature. For all specimen geometries, there was a temperature drop of 10 to 25°C when the specimens were moved from the water soak tank to the oven.

A brief temperature increase was observed in geometry C, following the specimens being placed into the vacuum vessel. This can be accounted for by the heat of wetting, which is an exothermic reaction when wood absorbs bound water in the cell wall. Since the heat of wetting is small, it is not often detected if sensible heat transport is rapid or the bound water adsorption rate is slow. This effect can be observed in Figure 4-22.

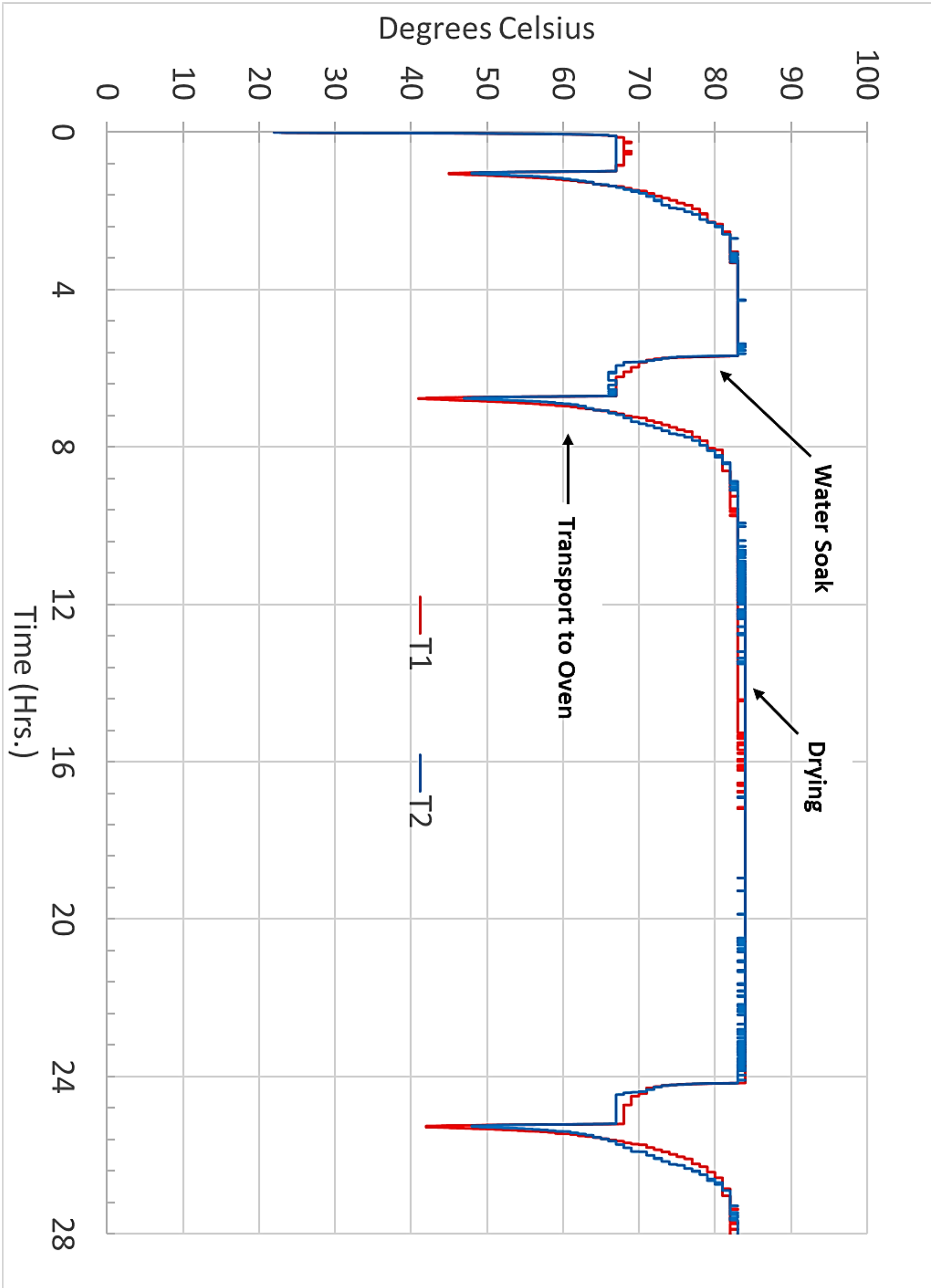

Figure 4-21, Bondline temperature results for Geometry T, 3 cycles VPS

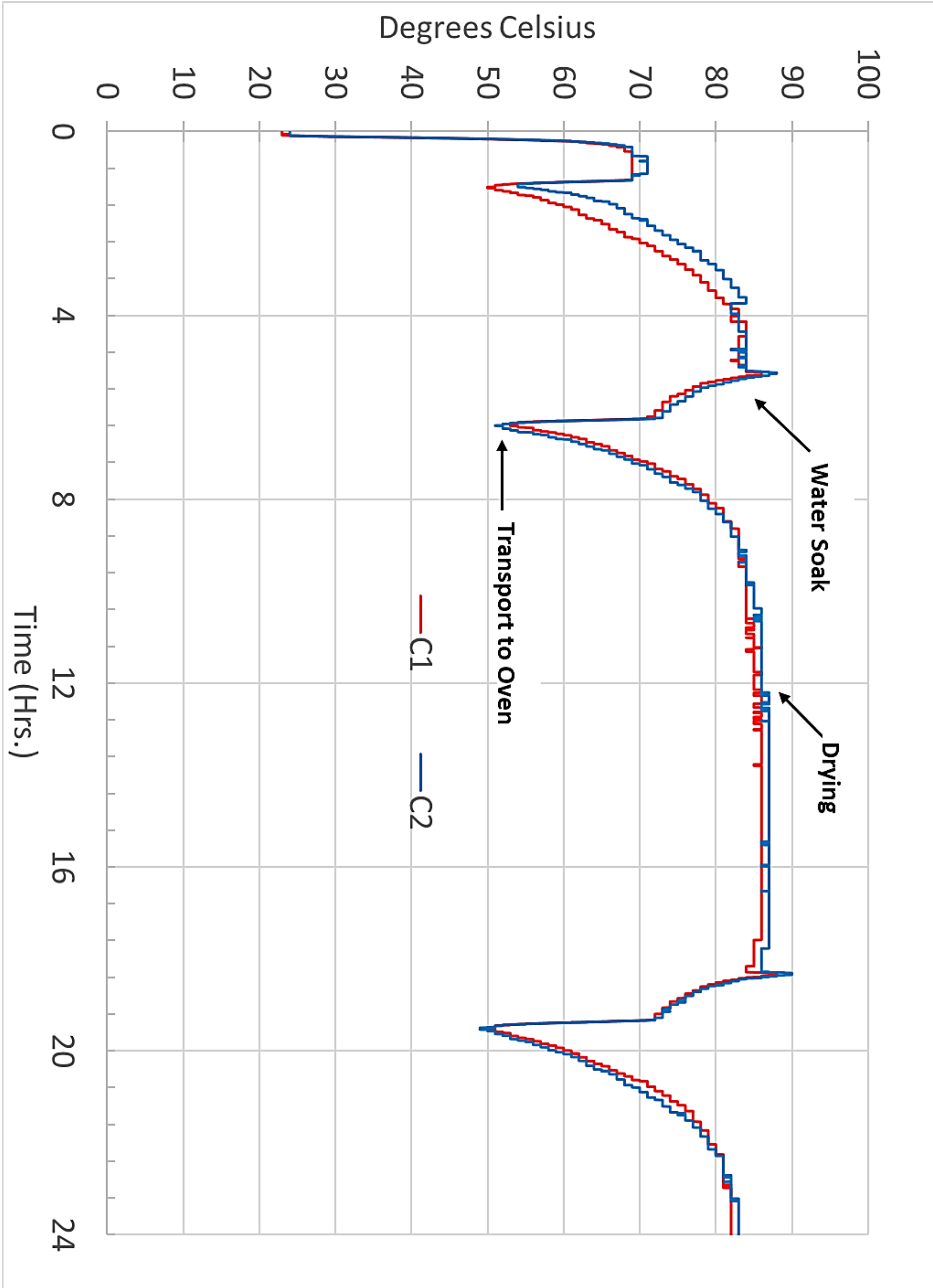

Figure 4-22, Bondline temperature results for Geometry C, 3 cycles VPS

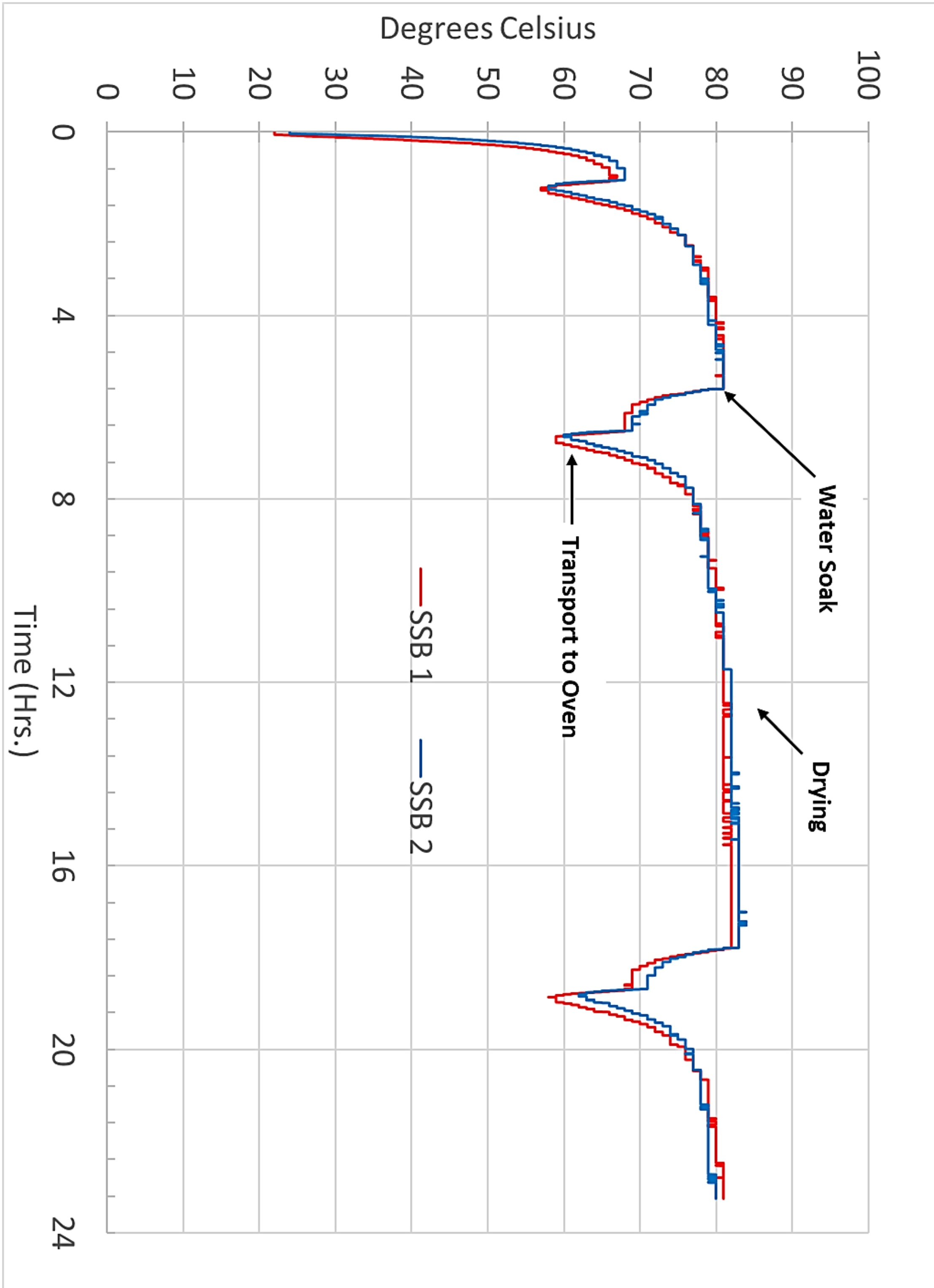

Figure 4-23, Bondline temperature results for Geometry SSB, 3 cycles VPS

4.3.3 CSA 0112.9 – BDF

A review of exposure conditions for the Boil-Dry-Freeze test are listed in Table 4-17.

Table 4-17, Exposure Conditions for the Boil-Dry-Freeze Test

CSA 0112.9 – Boil-Dry-Freeze
1. Boil *at least* 4hrs
2. Oven dry at 60°C for 19hrs
3. Freeze at ≤ -30°C for *at least* 4hrs
4. Repeat steps 1-3 for 8 total cycles

Similar to the VPS test, bondline temperature for all geometries responded to the cyclic conditions imposed by BDF exposure. This suggests that each phase within BDF cycles are adequate for all geometries in this study. There was a difference between the specimen geometries in regard to the temperature of the bondline in the oven. Geometries T and SBB had about the same heating-up time of 3 hours in the oven.

However, the SBB specimens started at about 52°C, while the T specimens started at about 38°C. Although the CSA standard specifies a drying phase of 19 hours, the actual drying times for the T, C, and SBB specimens were 24, 11, and 18 hours, respectively. Therefore, the results from this study must be considered a “modified” CSA 0112.9 standard, and the specimen geometries cannot be directly compared to one another. Nevertheless, the rate of change of temperature, as well as the steady-state temperature values, are noteworthy.

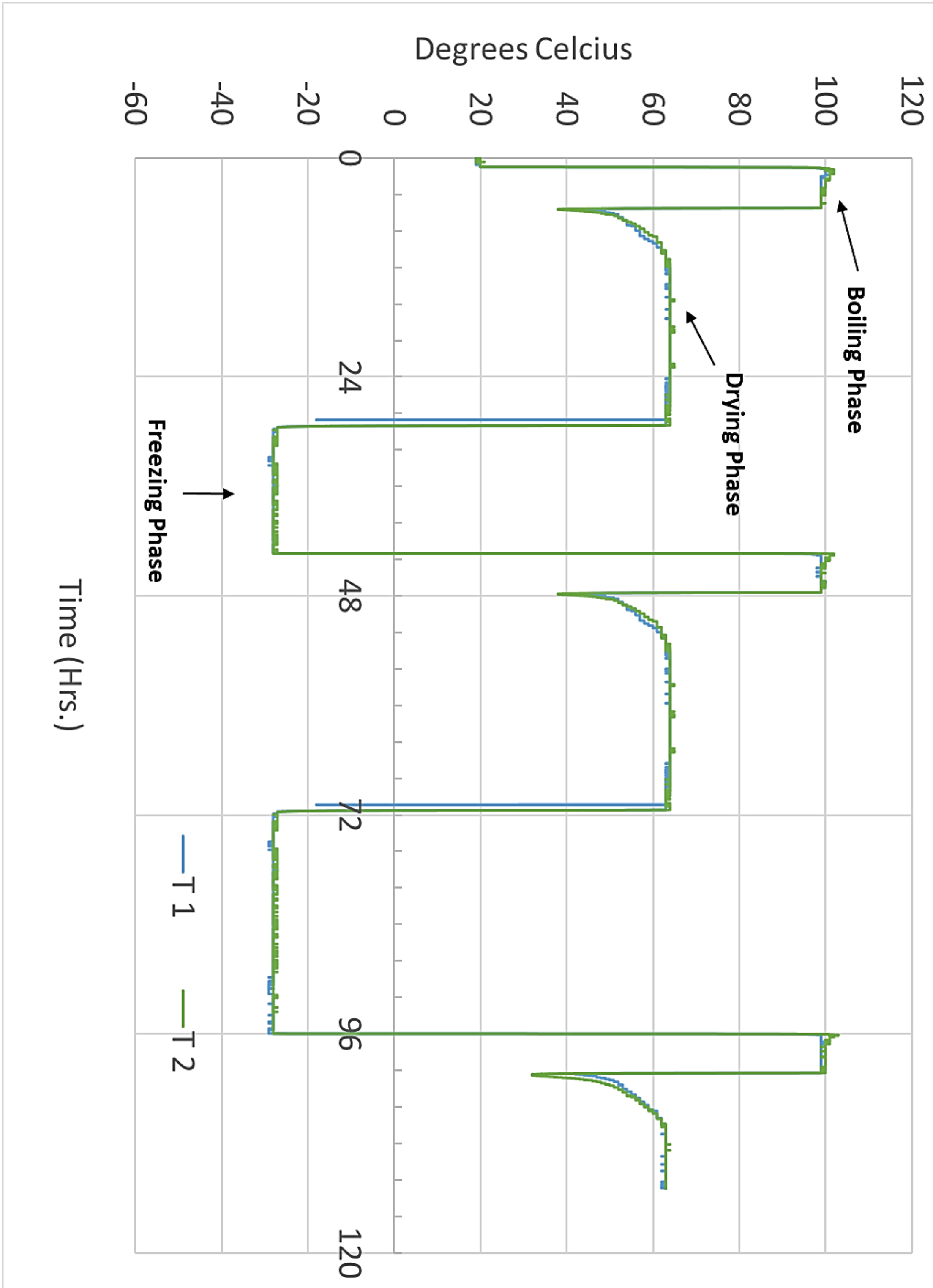

Figure 4-24, Bondline temperature results for Geometry T, 3 cycles BDF

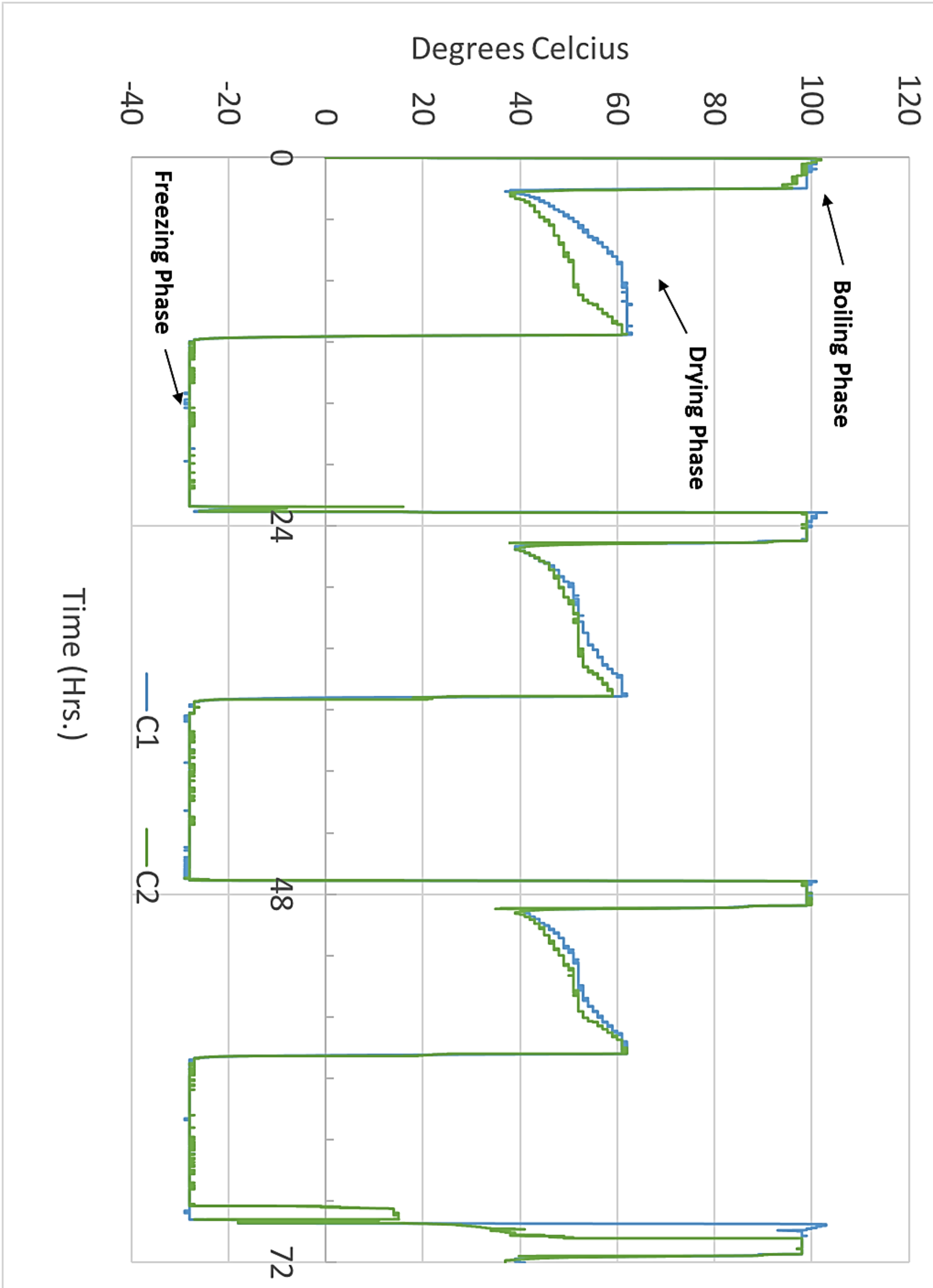

Figure 4-25, Bondline temperature results for Geometry C, 3 cycles BDF

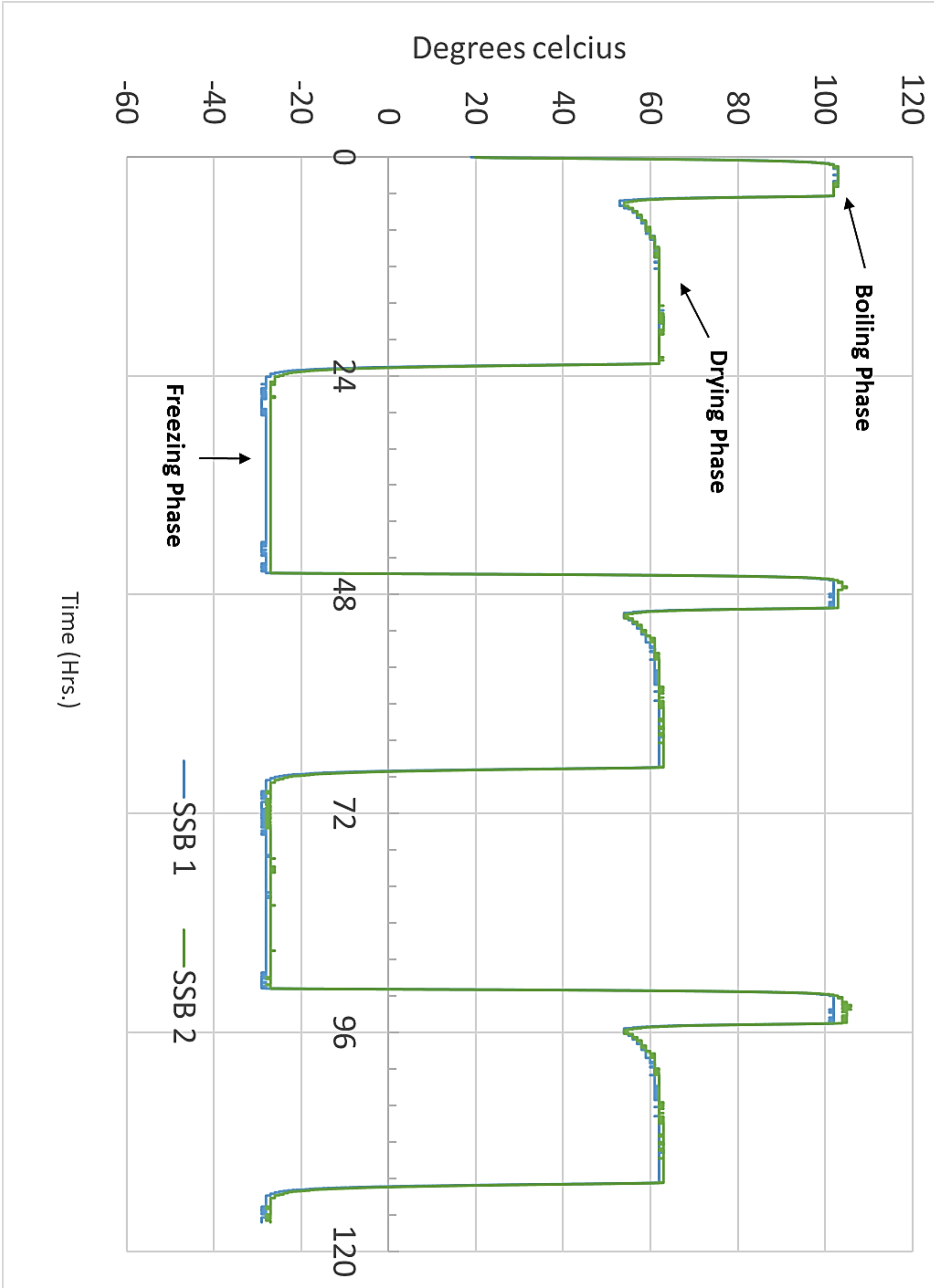

Figure 4-26, Bondline temperature results for Geometry SSB, 3 cycles BDF

4.4. Percent Wood Failure Analysis

There was no significant difference of percent wood failure (PWF) between the dry control specimens and the cyclic test specimens for the lap-shear in tension geometry and the compression shear block geometry. All specimens had at least 74% wood failure after exposure in the ABT (Tables 16 – 18). Likewise, there was no significant difference of PWF between T and C specimen geometries after exposure to either the BDF or VPS tests (Tables 19 and 20). These results indicate that the mechanical tests largely were an evaluation of the residual wood strength rather than adhesive bond strength. The specimen geometry had no influence on PWF, and the accelerated weathering protocol did not affect PWF.

Table 4-18, Summary statistics of percent wood failure, for geometry T: DC, WC, 400 cycles ABT

Cycle	N	Mean	StDev	95% CI
DC	10	84.50	13.22	(76.65, 92.35)
WC	10	93.00	6.32	(85.15, 100.85)
20	10	92.50	6.35	(84.65, 100.35)
40	10	81.82	13.83	(74.33, 89.30)
100	10	80.56	16.29	(72.28, 88.83)
200	10	85.00	15.81	(77.15, 92.85)
400	10	89.00	11.25	(81.15, 96.85)

Table 4-19, Summary statistics of percent wood failure, for geometry C w/ PF: DC, WC, 400 cycles ABT

Cycle	N	Mean	StDev	95% CI
DC	10	90.00	5.77	(82.33, 97.67)
WC	10	86.00	13.08	(78.33, 93.67)
20	10	82.00	14.57	(74.33, 89.67)
40	10	79.55	12.74	(72.23, 86.86)
100	10	85.56	8.08	(77.47, 93.64)
200	10	89.50	11.89	(81.83, 97.17)

400	10	84.50	15.36	(76.83, 92.17)

Table 4-20, Summary statistics of percent wood failure, for geometry C w/ PU: DC, WC, 400 cycles ABT

Cycle	N	Mean	StDev	95% CI
DC	10	79.50	16.74	(70.10, 88.90)
WC	10	82.50	13.18	(73.10, 91.90)
20	10	81.50	15.28	(72.10, 90.90)
40	10	80.55	12.78	(71.58, 89.51)
100	10	74.44	17.93	(64.53, 84.35)
200	10	81.50	14.73	(72.10, 90.90)
400	10	75.00	13.33	(65.60, 84.40)

Table 4-21, Summary statistics of percent wood failure, for geometry T, C: VPS

Geometry	N	Mean	StDev	95% CI
T	29	83.79	13.41	(79.43, 88.16)
C	29	90.52	9.76	(86.16, 94.88)

Table 4-22, Summary statistics of percent wood failure, for geometry T, C: BDF

Geometry	N	Mean	StDev	95% CI
T	30	84.48	13.72	(79.60, 89.36)
C	30	86.72	12.48	(81.85, 91.60)

5. Conclusions and Future Work

ABT results demonstrated a clear degradation curve for geometries C and SSB. For geometry T, future studies should consider increasing the number of specimens per group from 10 to 30. This would allow for a more robust statistical analysis of this geometry. All geometries subjected to the ABT experienced no drying throughout 400 cycles. As mentioned previously, this would imply that the dry phase of the ABT should be extended substantially to account for all AW geometries.

Additionally, separating specimens with spacers during active cycles, as Wilke (1974) performed, could allow adequate airflow around the specimens and in turn allow them to dry below FSP. The 10 min boiling phase allowed specimens from geometry T and C to promptly achieve boiling point. In contrast, geometry SSB did not realize boiling point throughout all 400 cycles. This would imply that larger specimens subjected to the ABT should not only have a longer dry phase but also an extended boiling phase.

Unlike the ABT, the VPS and BDF tests only require mechanical testing after the final cycle. Mechanically testing sample groups after each cycle from the BDF and VPS tests and comparing them to a dry control group of the selected geometry would allow them to be evaluated through non-linear regression, similar to the ABT. This would give industry insight on the effects of individual cycles on shear performance within geometry groups.

This study evaluated MC after each test’s corresponding dry phase. Future studies should account for MC measurements after each phase from all tests. For example, the BDF test would monitor MC after the boiling, drying and freezing phases. These data would be valuable if those conducting the test opted to extend the boil phase while holding the dry phase constant, as the standard allows. Extending the boil phase could have tremendous effects on the ability of specimens to dry below FSP. Considering that the freezing phase directly follows that of the boiling, specimens with higher moisture content may experience significant delamination due to the expansion of liquid water which effectively stresses the bondline.

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8. Appendix

Table 8-1, Raw values for percent wood failure - ABT

Cycle	TPF	%WF	CPF	%WF	CPU	%WF
WC	TPF-1	100	CPF-1	85	CPU-1	90
	TPF-2	90	CPF-2	90	CPU-2	75
	TPF-3	90	CPF-3	100	CPU-3	100
	TPF-4	100	CPF-4	85	CPU-4	40
	TPF-5	100	CPF-5	90	CPU-5	65
	TPF-6	85	CPF-6	85	CPU-6	80
	TPF-7	90	CPF-7	90	CPU-7	85
	TPF-8	100	CPF-8	85	CPU-8	85
	TPF-9	85	CPF-9	90	CPU-9	90
	TPF-10	90	CPF-10	100	CPU-10	85
DC	TPF-11	85	CPF-11	55	CPU-11	100
	TPF-12	90	CPF-12	90	CPU-12	100
	TPF-13	100	CPF-13	85	CPU-13	65
	TPF-14	55	CPF-14	80	CPU-14	65
	TPF-15	90	CPF-15	80	CPU-15	70
	TPF-16	90	CPF-16	95	CPU-16	75
	TPF-17	100	CPF-17	100	CPU-17	85
	TPF-18	75	CPF-18	100	CPU-18	90
	TPF-19	80	CPF-19	90	CPU-19	85
	TPF-20	80	CPF-20	85	CPU-20	90
20	TPF-21	85	CPF-21	90	CPU-21	85
	TPF-22	85	CPF-22	75	CPU-22	90
	TPF-23	100	CPF-23	55	CPU-23	100
	TPF-24	90	CPF-24	95	CPU-24	55
	TPF-25	95	CPF-25	100	CPU-25	90
	TPF-26	85	CPF-26	60	CPU-26	85
	TPF-27	90	CPF-27	85	CPU-27	80
	TPF-28	95	CPF-28	85	CPU-28	80
	TPF-29	100	CPF-29	85	CPU-29	95
	TPF-30	100	CPF-30	90	CPU-30	55
40	TPF-31	80	CPF-31	60	CPU-31	65
	TPF-32	90	CPF-32	75	CPU-32	75
	TPF-33	75	CPF-33	95	CPU-33	80
	TPF-34	55	CPF-34	90	CPU-34	90
	TPF-35	95	CPF-35	90	CPU-35	90
	TPF-36	100	CPF-36	100	CPU-36	100

	TPF-37	60	CPF-37	75	CPU-37	85
	TPF-38	85	CPF-38	80	CPU-38	86
	TPF-39	85	CPF-39	70	CPU-39	60
	TPF-40	85	CPF-40	65	CPU-40	65
100	TPF-41	90	CPF-41	75	CPU-41	90
	TPF-42	100	CPF-42	90	CPU-42	95
	TPF-43	75	CPF-43	95	CPU-43	95
	TPF-44	80	CPF-44	95	CPU-44	95
	TPF-45	50	CPF-45	80	CPU-45	75
	TPF-46	65	CPF-46	70	CPU-46	70
	TPF-47	75	CPF-47	80	CPU-47	65
	TPF-48	90	CPF-48	85	CPU-48	50
	TPF-49	95	CPF-49	90	CPU-49	50
	TPF-50	95	CPF-50	85	CPU-50	75
200	TPF-51	80	CPF-51	100	CPU-51	80
	TPF-52	55	CPF-52	100	CPU-52	80
	TPF-53	95	CPF-53	90	CPU-53	100
	TPF-54	100	CPF-54	100	CPU-54	95
	TPF-55	100	CPF-55	65	CPU-55	65
	TPF-56	85	CPF-56	85	CPU-56	65
	TPF-57	90	CPF-57	90	CPU-57	100
	TPF-58	90	CPF-58	90	CPU-58	90
	TPF-59	95	CPF-59	100	CPU-59	60
	TPF-60	60	CPF-60	75	CPU-60	80
400	TPF-61	75	CPF-61	55	CPU-61	85
	TPF-62	95	CPF-62	95	CPU-62	75
	TPF-63	95	CPF-63	100	CPU-63	75
	TPF-64	95	CPF-64	60	CPU-64	80
	TPF-65	65	CPF-65	85	CPU-65	65
	TPF-66	100	CPF-66	85	CPU-66	75
	TPF-67	90	CPF-67	85	CPU-67	90
	TPF-68	90	CPF-68	90	CPU-68	90
	TPF-69	100	CPF-69	90	CPU-69	70
	TPF-70	85	CPF-70	100	CPU-70	45

Table 8-2, Raw values for percent wood failure - VPS, BDF

PS2T	% WF	PS2C	% WF	CSAT	% WF	CSAC	% WF
PS2T-1	75	PS2C-1	90	CSAT-1	75	CSAC-1	80
PS2T-2	80	PS2C-2	80	CSAT-2	80	CSAC-2	85
PS2T-3	80	PS2C-3	100	CSAT-3	85	CSAC-3	85
PS2T-4	85	PS2C-4	100	CSAT-4	90	CSAC-4	100
PS2T-5	85	PS2C-5	100	CSAT-5	95	CSAC-5	90
PS2T-6	100	PS2C-6	100	CSAT-6	100	CSAC-6	95
PS2T-7	90	PS2C-7	90	CSAT-7	80	CSAC-7	85
PS2T-8	95	PS2C-8	90	CSAT-8	85	CSAC-8	90
PS2T-9	85	PS2C-9	100	CSAT-9	85	CSAC-9	95
PS2T-10	90	PS2C-10	100	CSAT-10	90	CSAC-10	100
PS2T-11	95	PS2C-11	85	CSAT-11	85	CSAC-11	100
PS2T-12	100	PS2C-12	90	CSAT-12	100	CSAC-12	65
PS2T-13	100	PS2C-13	100	CSAT-13	100	CSAC-13	65
PS2T-14	80	PS2C-14	85	CSAT-14	65	CSAC-14	90
PS2T-15	90	PS2C-15	90	CSAT-15	65	CSAC-15	85
PS2T-16	75	PS2C-16	85	CSAT-16	70	CSAC-16	90
PS2T-17	55	PS2C-17	90	CSAT-17	75	CSAC-17	95
PS2T-18	95	PS2C-18	100	CSAT-18	80	CSAC-18	100
PS2T-19	100	PS2C-19	55	CSAT-19	80	CSAC-19	100
PS2T-20	60	PS2C-20	90	CSAT-20	95	CSAC-20	80
PS2T-21	85	PS2C-21	85	CSAT-21	45	CSAC-21	90
PS2T-22	85	PS2C-22	80	CSAT-22	60	CSAC-22	75
PS2T-23	85	PS2C-23	80	CSAT-23	90	CSAC-23	55
PS2T-24	90	PS2C-24	95	CSAT-24	95	CSAC-24	95
PS2T-25	100	PS2C-25	100	CSAT-25	95	CSAC-25	100
PS2T-26	75	PS2C-26	100	CSAT-26	90	CSAC-26	60
PS2T-27	80	PS2C-27	90	CSAT-27	100	CSAC-27	85
PS2T-28	50	PS2C-28	85	CSAT-28	100	CSAC-28	85
PS2T-29	65	PS2C-29	90	CSAT-29	95	CSAC-29	95