CHAPTER 3: METHODS AND MATERIALS

3.1 Specimen Preparation

3.1.1 Specimen Evolution

In the original proposal, it was anticipated that the notched coating adhesion (NCA) specimen[6] would be used for this investigation. The NCA specimen would be used to measure the fracture toughness of the interface between the polymer concrete overlay and the aluminum substrate. Specimens were cut from initial sections provided by the Reynolds Metals Company. A notch was cut through the polymer concrete across the specimen to the aluminum substrate surface in order to create a characteristic NCA specimen. Preliminary testing suggested that the NCA type specimen would be suitable for this investigation. It was then requested that Reynolds fabricate a sufficient quantity of sections to obtain the specimens needed for this investigation.

Eight beam sections were received from the Reynolds Metals Company in March 1997. The beam dimensions were 305 mm [12 in] in width, 1.8 m [6 ft] in length and 305 mm [12 in] in height. The beam consisted of an aluminum top and bottom flange with webbing, and a polymer concrete overlay on the top flange. Figure 1 presents a cross-section of the beam section. The specimen dimensions were 305 mm [12 in] by 51 mm [2 in]; each specimen consisted of an aluminum substrate approximately 11 mm thick and a polymer concrete overlay that was approximately 9 mm thick. Figure 2 shows the specimen plan dimensions.

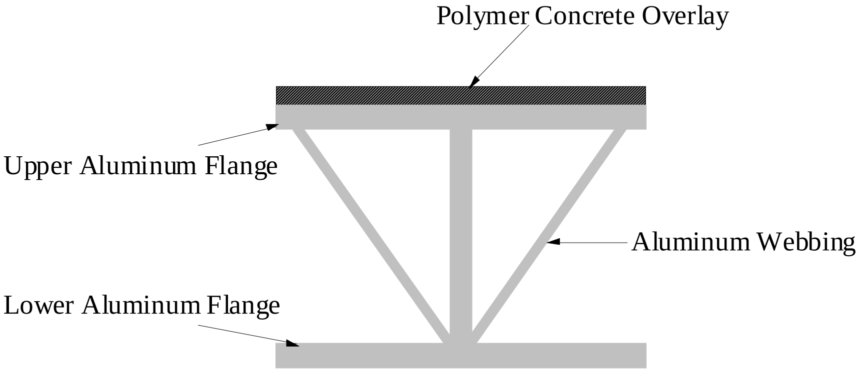
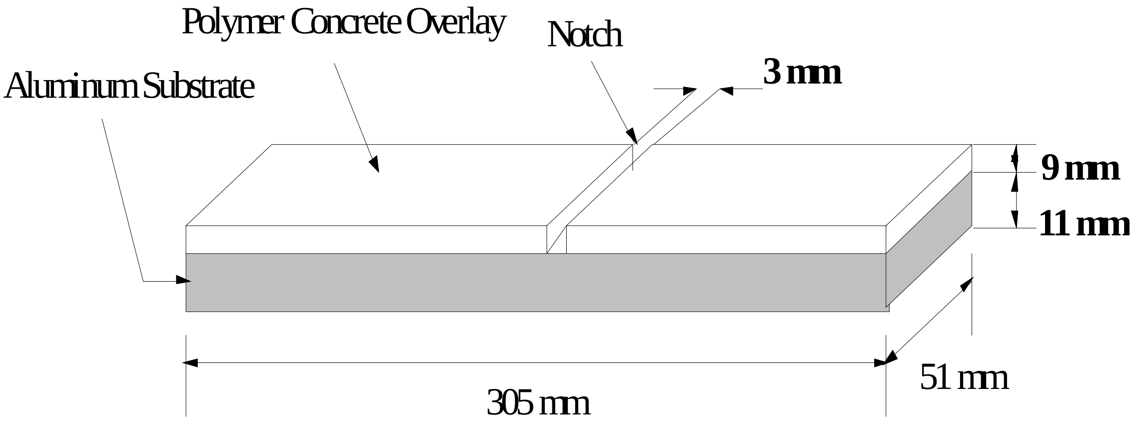


Figure 1: Cross-section of the beam section



.**Figure 2:** Specimen plan dimensions

Specimens were cut from the beam sections using a commercial water jet cutter, an abrasive was used in the water to cut through the polymer concrete overlay and the aluminum. This cutting process produced a smooth cut that appeared well suited for this material. Twenty four specimens were obtained from each beam section for a total of 168 specimens. All of the specimens were numbered in accordance with their location on the original beam sections. The numbers were etched on the aluminum surface of each specimen. A 3 mm notch was then cut at the center of each specimen through the polymer concrete overlay to the aluminum surface across the center of the specimen width. The notch was cut using an abrasive wheel. The notch produced a characteristic notched coating adhesion (NCA) specimen. After the specimens were numbered and notched, they were post cured in an oven at 60 ^{O}C [140 ^{O}F] for 24 hours. One of the reasons for the postcuring was to ensure that the specimens were fully cured. The postcure enhances the crosslinking in the polymer concrete overlay, this allows the material to be more stable. Another reason for the postcuring was that it would simulate what would occur to the overlay when exposed to hot summer temperatures when in service. After postcuring, some of the specimens were placed in the prescribed environmental conditions and allowed to age. The remaining specimens were used as control test specimens.

Subsequent testing on some of these specimens resulted in the overlay not debonding from the aluminum substrate. The specimen coating did not meet the coating thickness specifications. Some of these specimens had a coating thickness of as little as 4-5mm. Because of this reduced coating thickness, there was too little energy being stored in the overlay to produce a debond at the interface.

In order to salvage the specimens that had already been fabricated, a decision was made to alter the test specimens. It was decided that an aluminum plate would be bonded to the overlay surface in the hopes that it would provide sufficient energy storage to produce debonding at the interface between the overlay and the original aluminum substrate. These aluminum plates were cut in the same dimensions as the NCA specimens from the remaining scrap aluminum from the bottom flange. The aluminum plates were to be bonded to the NCA specimens using the same Tamms Flexolith 216R epoxy that was used in the overlay.

Two different pretreatments were tried on the bonding surface of the aluminum plates. The first method involved a chemical pretreatment. The second method involved scrubbing the bonding surface with a 200-grit sandpaper until a smooth, bright surface was produced. Subsequent testing showed that both pretreatments produced a satisfactory bond between the aluminum plate and the overlay. It was decided that the second method would be easier to use for pretreatment.

Pretreated aluminum plates were then bonded to some of the NCA specimens to create sandwich type specimens. Figure 3 presents the sandwich specimen.

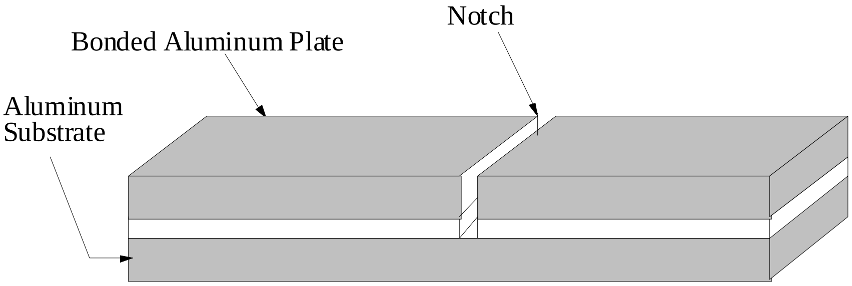


Figure 3: Sandwich specimen.

A decision was made by those in the Engineering Science and Mechanics Department to perform a mixed mode flexure (MMF)[7] bending test on these specimens to determine if this test method produced an interfacial failure between the overlay and the original aluminum substrate.

The MMF bending test was adopted to determine the critical strain energy release rate, which is a measure of the fracture toughness of the material. As the name implies, the interfacial debonding is under combined opening and shear mode loading. In a standard MMF test, the applied load is centered between the two supports[8]. Because of the dimensions of the specimens, it was decided that the applied load would be located at the quarter point of the specimen, thus producing a modified MMF test geometry. Some of the advantages associated with this modified MMF test are:

1. Plastic deformation of the aluminum substrate is avoided during testing.
2. Each specimen can be tested twice.
3. Only one debond propagates during testing.

Figure 4 presents the modified MMF test geometry.

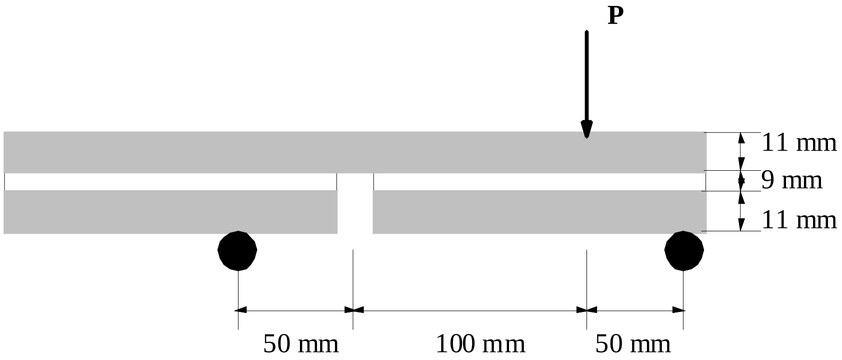


Figure 4: Modified Mixed Mode Flexure (MMF) Test Geometry.

An initial crack was introduced at the primary interface by using a three point bending test as shown in figure 5.

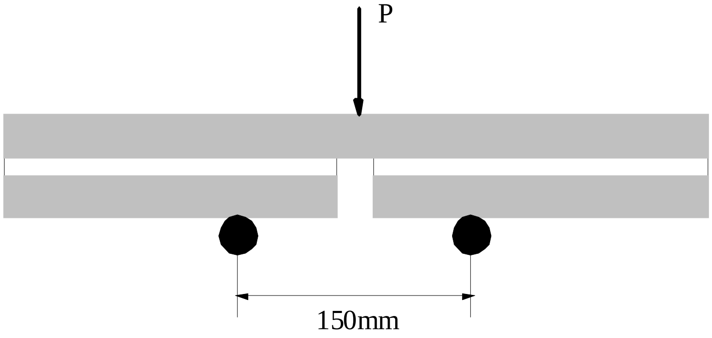


Figure 5: Precrack Test Geometry.

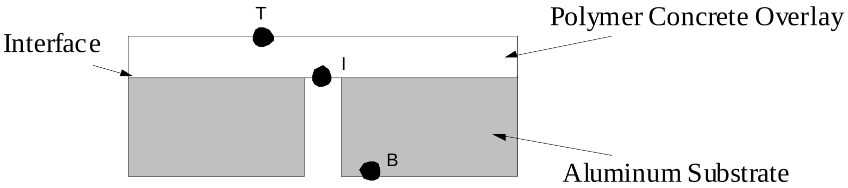
The postcured specimens performed better in the precracking test. It was easier to obtain an initial crack in the postcured specimens. These specimens appeared to be more brittle than the non-postcured specimens; this more brittle behavior was probably due to the increased crosslinking of the material during postcuring.

After the sandwich specimens had been precracked, they were tested using the modified MMF bending test. Preliminary testing of the specimens at room temperature showed that the applied load necessary to produce additional debonding increased as the crack grew. These results are the opposite of what would be expected, the critical load should decrease with increasing crack growth. It was suspected that the viscoelastic flow of the overlay was reducing the available strain energy release rate. In order to reduce the influence of viscoelasticity, it was decided that the test specimens would be conditioned in dry ice before testing. This enabled the specimens to reach a test temperature of approximately –33 ^{O}C [–27 ^{O}F]. This procedure of conditioning the specimens in the dry ice was later adopted for both the precracking and the modified MMF testing of the remaining specimens.

3.1.2 Outdoor Temperature Specimen

The remaining beam was used for an outdoor temperature experiment. The purpose of this experiment was to determine the maximum and minimum field temperatures that the polymer concrete and aluminum would achieve when exposed to a typical Virginia weather condition. The experiment was conducted from July 1997 to June 1998.

Copper/ Constantine thermocouples were attached at the top surface of the overlay, at the location of the interface between the overlay and the aluminum substrate, and at the bottom of the aluminum surface of the top flange. Figure 6 presents the thermocouple locations.



T: Top surface of the polymer concrete
I: Interface
B: Bottom surface of the aluminum substrate

Figure 6: Thermocouple location

The thermocouples were located at various sites on the beam section, with each site consisting of three thermocouples attached at the previously mentioned locations. Figure 7 presents a top view of the locations of these sites on the beam section.

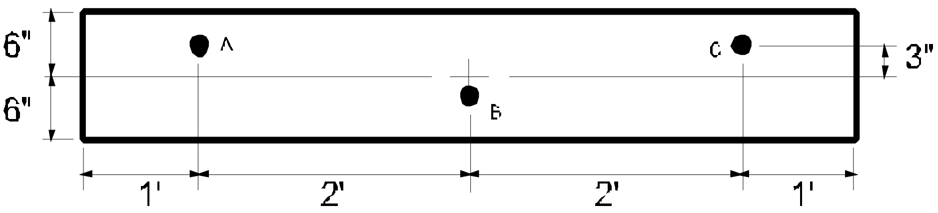


Figure 7: Thermocouple locations on beam section.

A 5 mm [3/16 in] hole was drilled through the aluminum to the interface from the bottom of the sample. The thermocouples were attached using an epoxy. The sides of the beam section were covered with 25 mm thick styrofoam sheeting in order to simulate in-service boundary conditions. The beam section was then placed outside on concrete blocks to elevate it off the ground about 1.2 m [4 ft]. The thermocouple wires were run to the data acquisition device through a PVC pipe in order to protect the wires. The data acquisition device was set to take temperature readings every 30 minutes.

3.2 Test Method

3.2.1 Strain Energy Release Rate

As mentioned previously, a modified version of the mixed mode flexure (MMF) test was used in this investigation. The modified MMF test was conducted on the specimens to determine the critical strain energy release rate, G_{cr}, which is a measure of the fracture toughness of the material.

The compliance method[9] was used to determine the critical strain energy release rate:

$$G = \frac{P^2}{2B}\frac{\partial}{\partial}\frac{C}{a} = -\frac{1}{B}\frac{\partial}{\partial}\frac{U}{a} \qquad \text{(Equation 1)}$$

Where: P = force at the onset of crack growth

a = crack length

b = specimen width

C = compliance

U = strain energy stored in the specimen

A load vs. displacement plot was produced for each side of each specimen tested Compliance values were obtained from these plots. Figure 8 presents a typical load vs. displacement plot for a specimen tested in this investigation. This plot was generated by Huiying Zhang. An average compliance value was calculated from the plot using:

$C = \frac{y}{P}$ Where: y = displacement, P = load

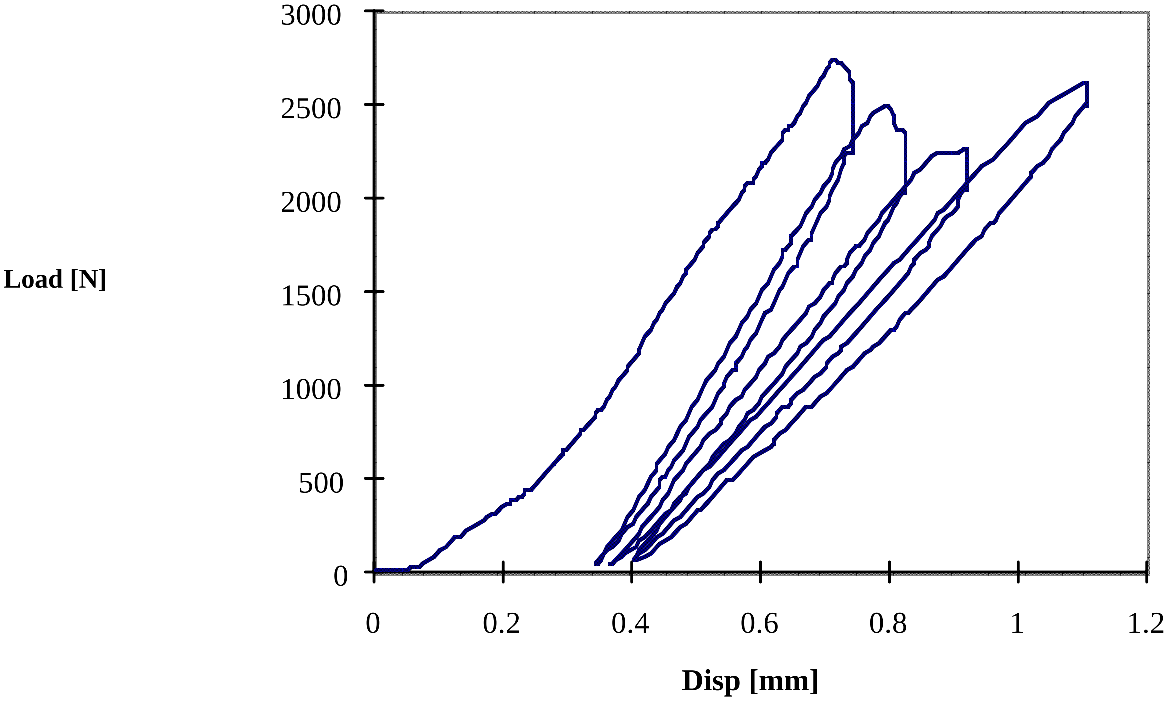


Figure 8: Load vs. Displacement Plot

The specimens were tested in groups; for example, all of the specimens exposed to six months of salt water soak conditioning, were tested in one group. Load vs. displacement plots were produced for each side of each specimen. Compliance values were calculated for each side of each specimen and these values were plotted on a cube root of compliance ,$C^{1/3}$ vs. crack length, a. This $C^{1/3}$ vs. a plot was for the entire group of specimens tested. Equation 1 can then be used to determine the critical strain energy release rate for each side of each specimen in the group. Figure 9 presents a typical cube root of compliance, $C^{1/3}$ vs. crack length, a plot. This Plot was also generated by Huiying Zhang.

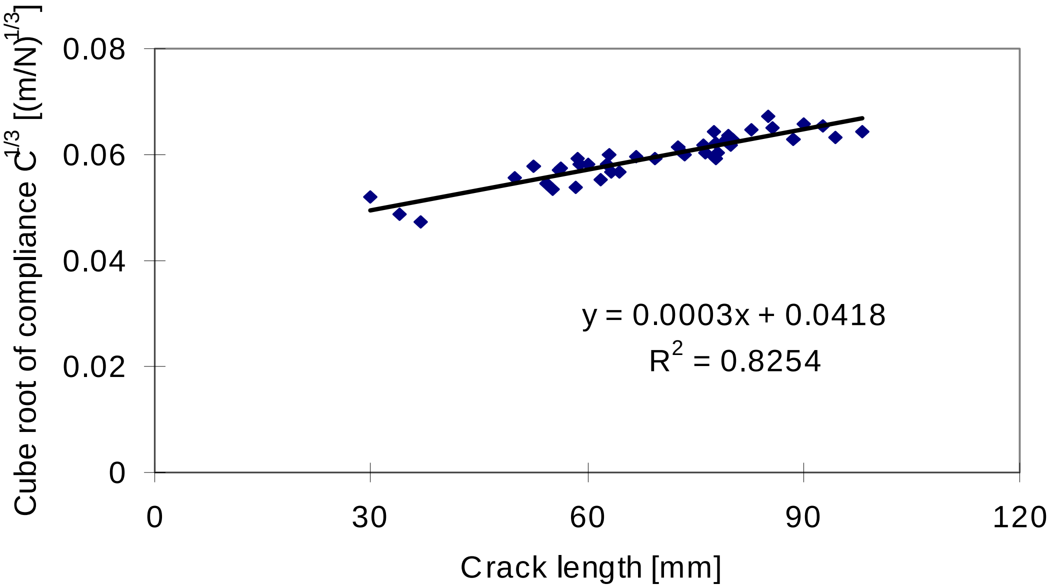


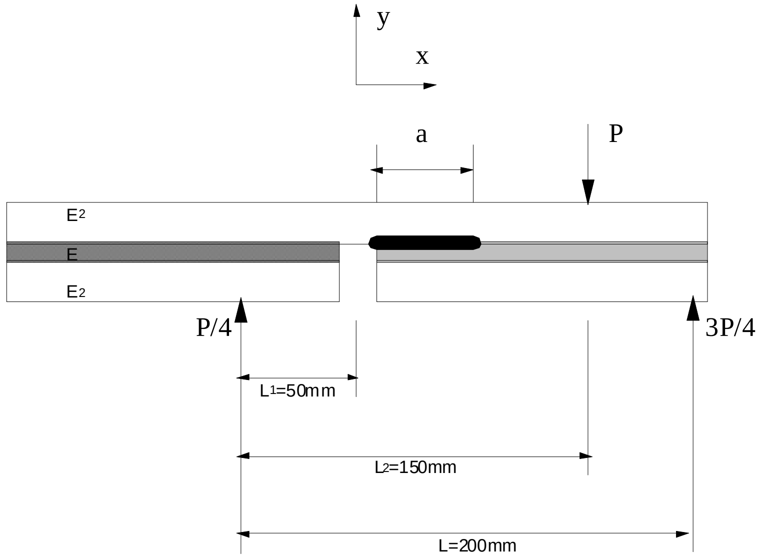
Figure 9: Cube Root of Compliance vs. Crack Length Plot

A linear elastic fracture mechanics solution can be derived analytically from beam theory. The bending strain energy[10] in beams is:

$$U = \int (M^2 / 2EI)\, dx$$

where the integration is carried over the length of the beam.

Huiying Zhang derived an analytical solution for the test specimen geometry used in this investigation [11]. The following presents the test specimen geometry used in this solution.



$$U = \int_{-L_1}^{0} \frac{1/4\,P^2 x^2}{2E_2 I}\,dx + \int_{0}^{a} \frac{1/4\,P^2 x^2}{2E_2 I_2}\,dx + \int_{a}^{L_2} \frac{1/4\,P^2 x^2}{2E_2 I}\,dx + \int_{L_2}^{L} \frac{3/4\,P^2 (L-x)^2}{2E_2 I}\,dx$$

$$G = -\frac{1}{b}\frac{\partial U}{\partial a} = \frac{P^2(L_1+a)^2}{32bE_2}\left(\frac{1}{I_2}-\frac{1}{I}\right)$$

where: U = strain energy stored in the specimen

G = strain energy release rate

a = initial crack length

P = load

E_2 = elastic modulus of the aluminum

I = moment of inertia of the sandwich beam

I_2 = moment of inertia of the aluminum beam

3.2.2 Test Procedure

As stated previously in section 3.2.1, the modified MMF test is conducted to determine the critical strain energy release rate, G_{cr}, which is a measure of the fracture toughness of the material. The first part of the test involved placing the specimen in a dry ice to obtain a temperature of -33 ^{O}C [-27 ^{O}F] to reduce viscoelastic influences. After reaching temperature, the specimen was placed in a three point bend test to initiate a precrack at the polymer concrete and aluminum substrate interface. The precrack length was measured on each side of the notch in the specimen. The specimen was then placed back in the dry ice to return the test specimen temperature to -33 ^{O}C [-27 ^{O}F]. The specimen was removed from the dry ice and the MMF test was conducted on one side of the specimen. The specimen testing was conducted on an Instron 4505 universal test frame controlled by a LabVIEW program, (LabVIEW is a software developed by National Instrument). The specimen was monitored during loading, the load was held when crack propagation started. The crack length was measured and recorded. The specimen was then unloaded to complete one cycle. Three to five cycles were completed on each side of each specimen tested. The specimen was then returned to the dry ice and the second side of the specimen was then tested after the specimen had returned to the test temperature of -33 ^{O}C
[-27 ^{O}F].

3.3 Environmental Exposure Conditioning

3.3.1 Humidity Chamber Conditioning

The humidity chamber conditioning involves three different temperatures at 98 % relative humidity. The three temperatures were 30 ^{O}C [86 ^{O}F], 45 ^{O}C [113 ^{O}F] and 60 ^{O}C [140 ^{O}F]. The relative humidity (RH) was measured with an RH probe. The probe was inserted into the one inch diameter opening in the top of the humidity chamber and the corresponding RH value was recorded. The temperature was monitored by thermometers that were placed in each humidity chamber at the level of the specimens. The temperature was checked each day to ensure that the temperature remained at the prescribed test temperature. If the temperature deviated from the prescribed temperature, it was adjusted with the temperature control on the humidity chamber. Deionized water was put into each of the three humidity chambers to a level of approximately 51 mm [2 in]. A support made of chlorinated poly-vinyl chloride (CPVC) pipe was placed into each chamber, the supports were approximately 76 mm [3 in] in height. The purpose of the supports was to keep the specimens above the water level and in the 98% relative humidity condition. Two aluminum strips were placed across each support in order to add extra support for the weight of the specimens. The specimens were stacked directly on the supports in layers. Each layer consisted of 4 specimens. Between each layer, two plexiglass strips were placed in order to prevent the specimen layers from touching. This also allowed moisture to enter the overlay surface of the specimens. The specimens layers were rotated on a weekly basis to allow for uniform conditioning of all specimens. Twenty (20) specimens were conditioned in each chamber.

3.3.2 Freezing and Thawing

Specimens for freezing and thawing (F&T) conditioning were conditioned according to the following cycle:

60 ^{O}C [140 ^{O}F], 100% RH or 20 hours
-25 ^{O}C [-13 ^{O}F] for 4 hours

The specimens were placed in a polypropylene container. The container was filled with deionized water until the specimens were completely submerged. The container was then covered and placed in an oven at 60 ^{O}C [140 ^{O}F] for 20 hours. After 20 hours, the specimens were removed from the container and placed in the freezing and thawing chamber. The freezer plate in the freezing and thawing chamber was covered with a 4 mil plastic sheet, the specimens were then placed on the plastic sheet. The specimens were then covered with another 4 mil sheet of plastic in order to keep them moist. The specimens were allowed to cool for 4 hours. After 4 hours of cooling, the specimens were placed back into the polypropylene container, covered and placed back into the oven. This completed one cycle. The specimens were moved between the oven and freezer each day, with one cycle taking one day to complete. Twenty (20) specimens were conditioned.

3.3.3 Salt Water Soak

The specimens were conditioned in a 6% NaCl (by weight) solution. The specimens were placed in a polypropylene container. The container was then filled with 6% NaCl solution until the specimens were completely submerged. The container was covered and placed in an oven at 60 ^{O}C [140 ^{O}F]. An air pump was attached to the container in order to aerate the salt solution. The specimens were removed from the container once per week and hand dried. The hand dried specimens were then placed in a clean, dry polypropylene container and placed back into the oven at 60 ^{O}C [140 ^{O}F] for 24 hours. After 24 hours of drying, the specimens were placed back into the container filled with the 6% NaCl solution, covered and placed back into the oven. The specimens were dried on Tuesday of each week. Potential measurements (mV) were taken for 0, 4, 5, 6, 7, and 8% (by weight) NaCl solutions. Each solution was tested using a specific ion chloride electrode and the resulting potentials were recorded. A solution concentration or calibration curve was developed. For instance, a 6% solution had a - 85 mV potential and an 8% solution had a - 95 mV potential according to the developed curve. A potential measurement was then taken on the actual conditioning solution using the same electrode and the mV reading was checked against the developed curve to determine the actual solution concentration. A solution concentration curve was developed for each weekly assessment of the concentration of the conditioning salt solution. Twenty (20) specimens were conditioned.

3.3.4 Dry Conditioning

Twenty (20) specimens were dry conditioned. This procedure involved placing the specimens directly into an oven at 60 ^{O}C [140 ^{O}F] and allowing them to continuously dry.

The humidity chamber, salt water soak and dry conditioning test specimens were conditioned for one year. The freezing and thawing specimens were conditioned for 300 cycles. Table 1 presents the test matrix for the conditioned specimens as well as the control specimens.

Table 1: Study Test Matrix

		30^{O}C [86^{O}F], 98% RH	45^{O}C [113^{O}F], 98% RH	60^{O}C [140^{O}F], 98% RH	Freezing & Thawing	Salt Water Soak	Dry
Baseline (Control)	25						
Static Bond Toughness							
Test at 2 Months					6	6	
Test at 6 Months		7	7	7	7	7	8
Test at 9 Months		7	7	7			
Test at 12 Months		6	6	6	7	7	12

NOTE: The Control specimens were post-cured for 24 hours at 60^{O}C [140^{O}] before testing. All other specimens were post-cured in the same manner before conditioning.

3.4 Materials

3.4.1 Aluminum

The aluminum used for this system was an aluminum alloy 6063-T6 extruded bar stock. The aluminum alloy made up the top and bottom flange as well as the webbing of the beam section. This material generally possesses the following mechanical properties[12]:

Modulus of Elasticity: 70 GPa
Tensile Strength: 260 MPA
Coefficient of Thermal Expansion: $24 \text{ x } 10^{-6} \text{ /}^{O}\text{C}$

4.4.2 Polymer Concrete

The overlay for this system was an epoxy based polymer concrete. The epoxy was Tamms Flexolith 216 R. This was a two component epoxy compound. Part A was the base and Part B was the hardener. Part A contained ethylhexyl glycidyl ether and part B contained diethylenetriamine[13]. The two parts are combined 1:1 by volume and thoroughly mixed at a slow mix speed. The silica sand aggregate was mixed into the epoxy mixture, with the aggregate to epoxy binder ratio being 2.35:1. The mixture was then spread onto the aluminum panel. The technical data sheet from Tamms for Flexolith 216 listed the following mechanical properties for the material:

Modulus of Elasticity: 900 MPa
Tensile Strength: 19 MPa
Coefficient of Thermal Expansion: $9 \text{ x } 10^{-5} \text{ /}^{O}\text{C}$