DESIGN VALIDATION PLAN

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CONTENTS

1. REVISION HISTORY

Rev	**Description of Change**
A	First revision

2. APPROVALS

Approver	**Project Role**

3. ACRONYMS

LAr …. liquid argon

GAr … gas argon

BCR … Project baseline change request

EDMS … CERN document control system

DocDB … Fermilab document control system

4. INTRODUCTION

This document describes the test validation plan for a composite window on DUNE LAr membrane cryostats in the United States. The composite window is a new technology for LAr designs, as previous designs have been constructed of entirely steel framed. This test plan shall validate the composite window as a newer technology by executing multiple test processes.

4.1 CRYOSTAT DESCRIPTION

The LAr membrane cryostats are made of a stainless steel cold vessel that contains the cryogens, sensors, insulation panels and a warm steel supporting structure.

The cold primary membrane tank is made of a stainless-steel liner that contains the cryogenic liquid and gas. The liner maintains the leak tightness. The membrane liner is corrugated to provide strain relief, resulting from temperature related expansion and contraction.

The insulation is composed of layers of polyurethane providing a thermal barrier between the membrane at the liquid cryogen temperature and the support structure at ambient temperature. The secondary barrier located at the end of the insulation is a physical protection that contains the liquid cryogen in case of a failure of the first membrane.

The surrounding cryostat warm steel structure support consists of a frame of steel columns and beams. The frame is reinforced locally with corrugated plates. On one side of the cryostat, a composite window replaces the steel structure in order to limit the impact on crossing neutrinos. The window is connected to the steel beams with a scarf joint. The composite window also replaces the secondary barrier, and will provide leak and gas tightness.

An overview of the cryostat and a cross section are shown in Figure 1.

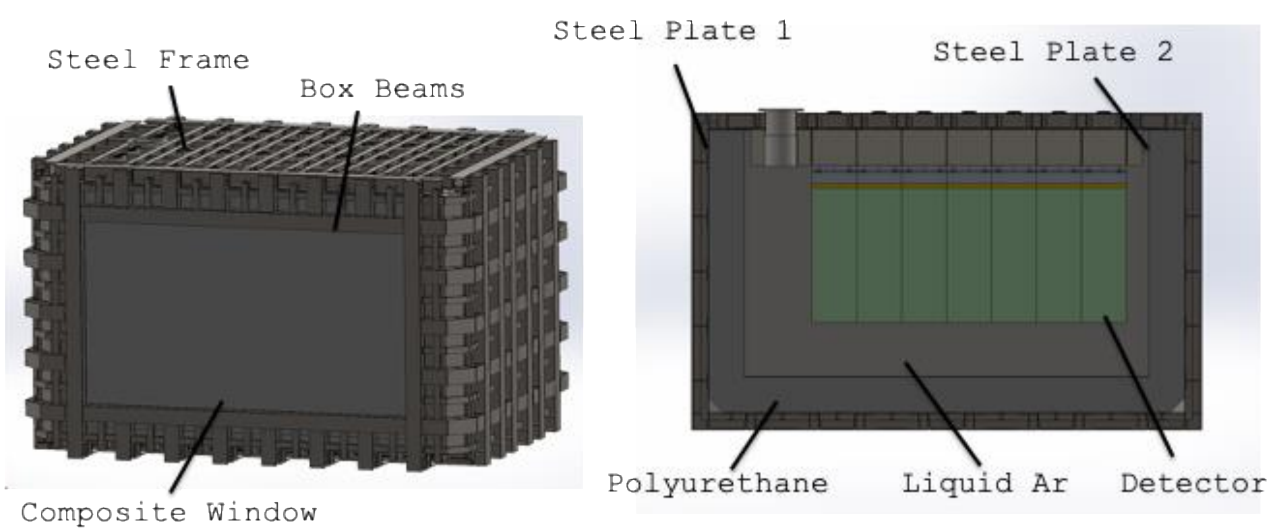

Fig. 1: Cryostat support structure overview (left) and cross-section (right).

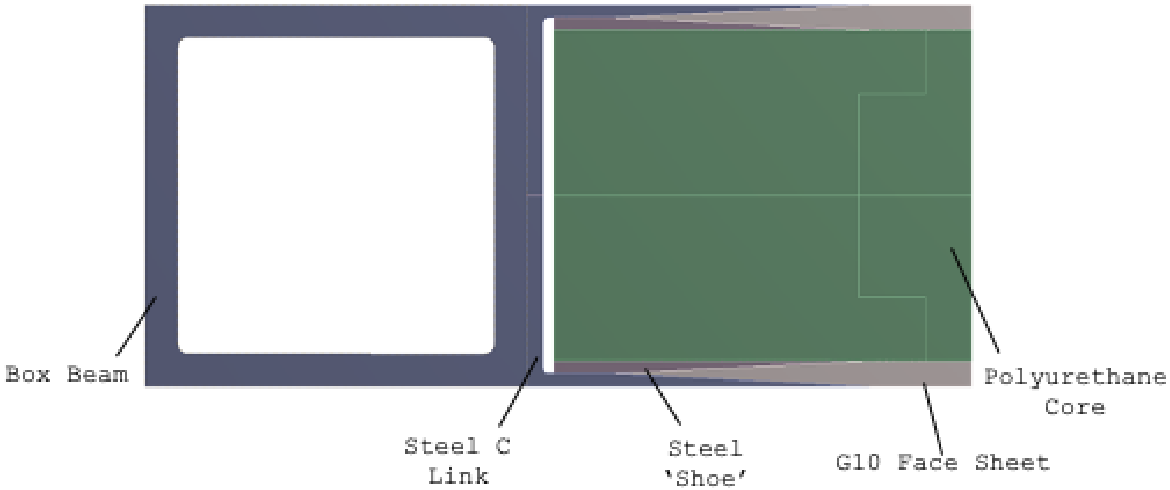

Fig. 1: Scarf joint connecting the steel structure to the composite window.

4.2 TEST PLAN SUMMARY

The test plan for the composite window will scale in complexity and scale.

Tests will start with validating the materials chosen and the scarf joint which is a critical element joining the composite window to the steel structure of the cryostat. Due to the size of the window the materials chosen will need to be validated to ensure the fabrication process will be successful.

After the scarf joint is validated, tests will move onto sandwich beam tests. Tests will be performed to understand cantilever bending strength in the sandwich. Additionally, there are a significant amount of non-mechanical tests to be performed with these samples that will assist in integration.

Finally a composite plate that closely reassembles the real window, but on smaller scale will be fabricated. This will be put under pressure for gas tightness and hydrostatic loading.

As such, these tests are split into 4 main categories. MATERIAL, JOINT, BEAM, and PLATE

1. **MATERIAL TESTS**
   A. RTM (VARTM) FLOW LENGTH & TIME VALIDATION
2. **JOINT TESTS**
   B. TENSILE COUPON TEST
      Fabricated and Tested in accordance to ASTM D3039
   C. COMPRESSION COUPON TEST
      Fabricated and Tested in accordance to ASTM D3410
   D. TENSILE COUPON TEST AT COLD
      Fabricated and Tested in accordance to ASTM D3039 near LN2 temperatures
   E. COMPRESSION COUPON TEST AT COLD
      Fabricated and Tested in accordance to ASTM D3410 near LN2 temperatures
3. **BEAM TESTS (Full Thickness)**
   A. CANTILEVER BEAM LOADING
   B. WELDING PROXIMITY INTEGRATION TEST
   C. THERMAL CYCLING
   D. OPTIONAL: FLAMABILITY
4. **PLATE TESTS (Scaled Thickness + Size)**
   A. PRESSURE LOAD TEST

5. SAMPLE SIZES

EN 1990:2002 Annex D provides specifications for sample size. Specifically Table D1 that describes *n* samples as they relate to known and unknown values.

Table 1. Table D1: Values of Kn for 5% characteristic value (From EN 1990:2002)[4]

n	1	2	3	4	5	6	8	10	20	30	∞
V_x Known	2.31	2.01	1.89	1.83	1.80	1.77	1.74	1.72	1.68	1.67	1.64
V_x Unknown	-	-	3.37	2.63	2.33	2.18	2.00	1.92	1.76	1.73	1.64

As shown there are diminishing returns on known vs unknown values as more samples are tested. The base sample size for tests in this document will be 6 samples per test.

6. MATERIAL VALIDATION

6.1 RTM LENGTH & TIME TEST (VALIDATE CHOSEN MATERIALS)

There are key considerations for material when fabricating a window of this size. Firstly the size itself is very large which means fabricating inside an autoclave in one-piece is near impossible. This means it needs to be made outside an autoclave and oven, thus a room temperature cure. Additionally due to the size the RTM method must be validated to allow resin to penetrate the entire length of the window. As seen in the figure below, if resin does not travel sufficient distance in the cure time then the part does not become fully wetted. The most common civil engineering works for composites will be performed by pultrusion or vacuum assisted resin transfer molding (VARTM / RTM)

Figure 1. Different RTM test samples showing resin travel over cure time

Ideal manufacturing process

- Room Temperature Cure
- Not limited by size (Does not need to fit in autoclave or oven)
- Slow Cure (to allow resins to fully transfer through fibers)
- Can be manufactured without significant equipment (does not require specialized facility)

The composite window can be split in a few main subcomponents.

Fiber: The fiber is expected to be glass fiber. However many different specifications exists for a recommended type of fiberglass. E-glass or R-glass fibers. Additionally this fiber should have very high fiber aerial weight to increase thickness quickly without excess number of layers. (25mm thickness ~ 100 layers at 0.25 per layer)

Resin: Resins should be room temp cure with a long pot life. Tg exceeding 60C.

- TotalBoat

- Entropy
- West Systems
- Hexion

Epoxy Resins will likely be a more applicable choice. Although Polyester and Vinyl Ester resins are recommended in many specs, they are unlikely to meet our requirements.

Core: Core should have a density of 90kg/m^3 or higher. E= 10MpA. V = 0.5 (incompressible). Balsa wood is the base material.

7. SCARF JOINT VALIDATION

7.1 TENSILE PROPERTIES

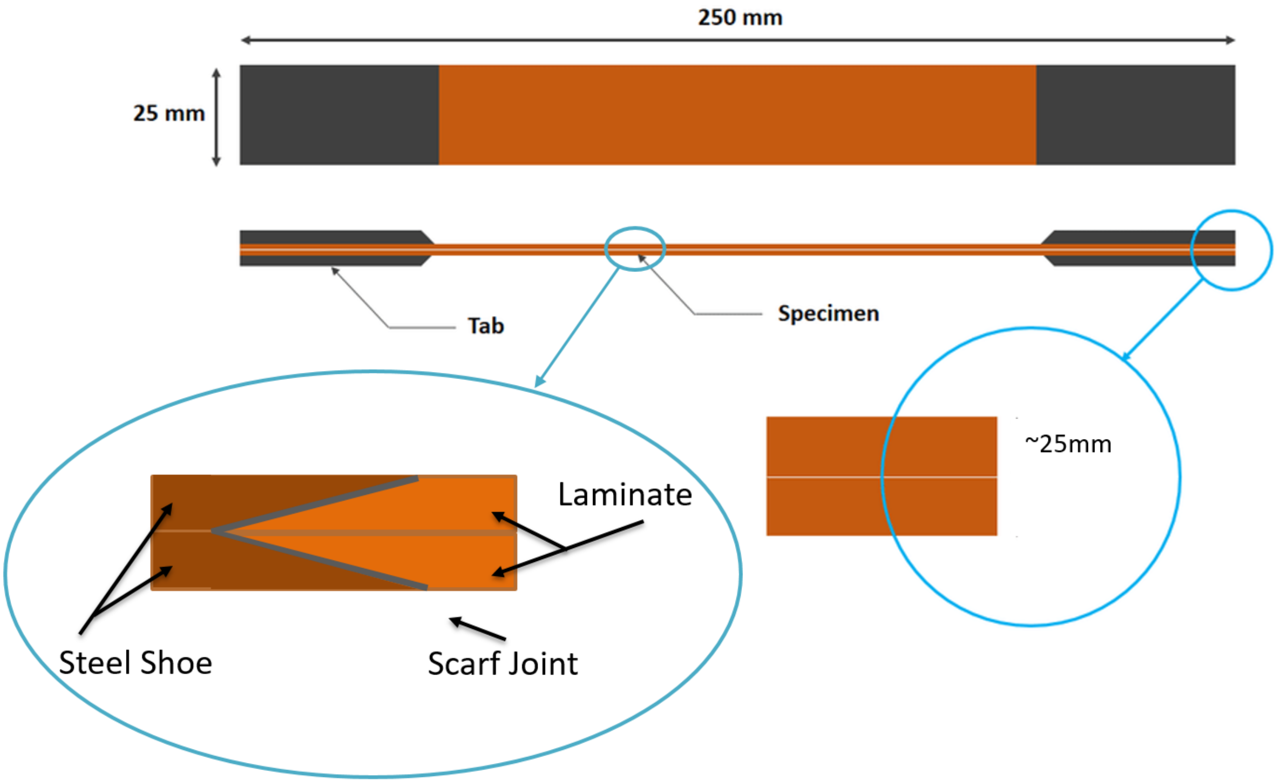

Figure 2. Coupon dimensions

Coupon dimensions are derived from ASTM standards. The non-standard feature being tested is the scarf joint within the laminate.

Complete testing guidelines for tensile properties of composite coupons are described by ASTM *D3039 Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials*. The test standard defines length and width requirements for the samples. A common recommended size for these samples are 250mm by 25mm, with 25mm on each end being a bonded metal tab. The complete details of this test are not be transcribed into this test plan. However, the general test process is:

1. Create standardized test coupons
2. Bond metallic tabs to the coupons. This reduces possible ‘crushing’ of the fibers when put into a universal test machine.
3. Apply tensile force using test machine. The machine will measure deflection and load applied.
4. Save stress/strain curve and load/deflection values.

The fracture load is of specific interest to us. We expect this load to exist within the laminate and not the joint.

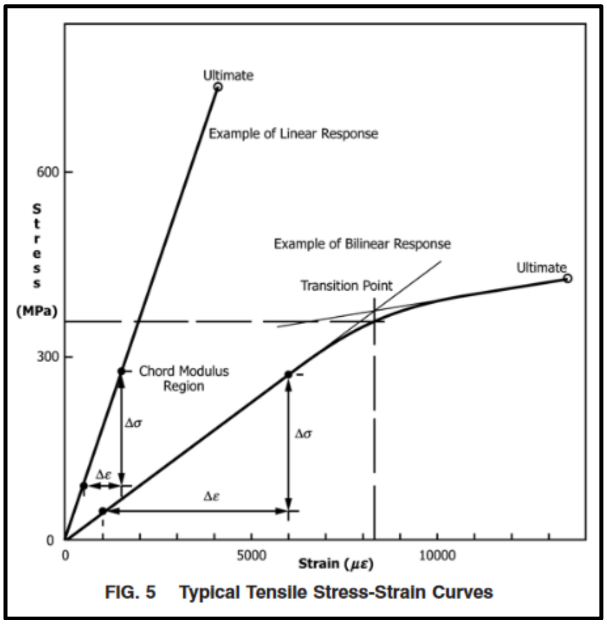

Figure 3. Example Stress/Strain curve from ASTM D3039

7.2 COMPRESSION PROPERTIES

Coupon sized for compression testing are slightly different. Width is still 25mm, length ~150mm with tabs of 65mm.

The complete test standard is contained within ASTM *D3410 Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading*[2]. The complete details of this test are not be transcribed into this test plan. The goal of the test is much the same as the tensile test, which is to develop a stress-strain curve.

7.3 TENSILE PROPERTIES AT COLD TEMPERATURE

Tensile properties at cold temperature follow the exact same standards are the Tensile Test. However, test samples will be subjected to load at near LN2 temperatures. This can be done using a thermal chamber, LN2 bath while under load, or have the test specimens removed from a LN2 bath then quickly inserted into the test setup.

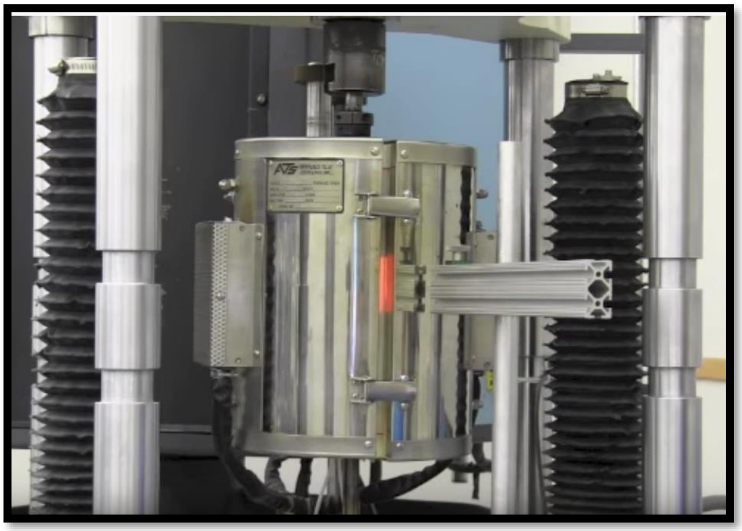

Figure 4. Universal Test Machine with thermal chamber

There are thermal chambers available that can provide hot or cold temperatures to samples while under load. Ideally the test provider will have a chamber unavailable. Understandably this equipment requires significant investment. As such, LN2 bath would also be sufficient to provide cold temperatures.

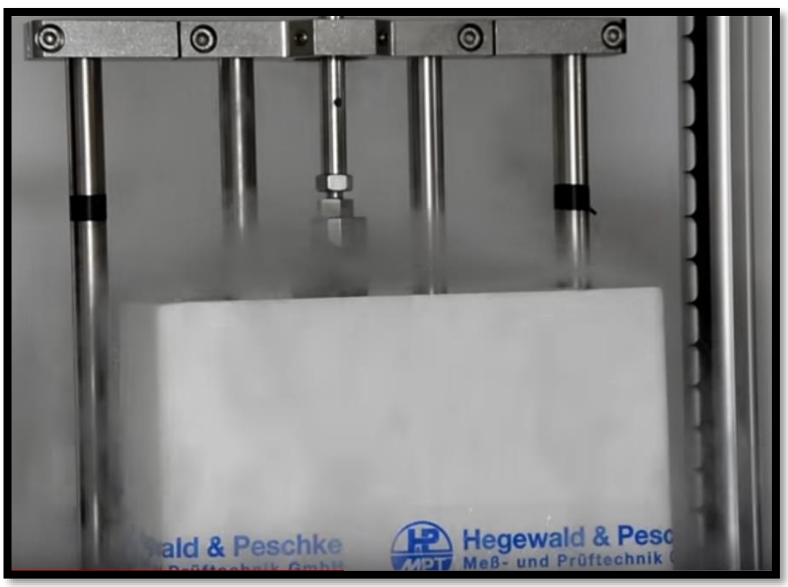

Figure 5. A LN2 bath installed to an Universal Test Machine

There are strategies to surround the sample in LN2 bath while under load. However, even simpler is to soak the test samples prior to the test and simply monitor temperature while load is applied. This strategy should be agreed upon with safety committees.

7.4 COMPRESSION PROPERTIES AT COLD TEMPERATURE

The compression test at cold is exactly the same as the D3410 compression standard, however including cold bath strategies described in the previous section.

7.5 LAMINATE PROPERTIES – NO SCARF JOINT

The Laminate-only properties will be critical for comparison of strength to test coupons that contain the scarf joint. The same tests will be performed using a series of laminate-only coupons that do not contain the scarf joint

8. SANDWICH BEAM PROPERTIES

8.1 MANUFACTURING

The Sandwich Beam samples will be manufactured as full thickness, but reduced width and length.

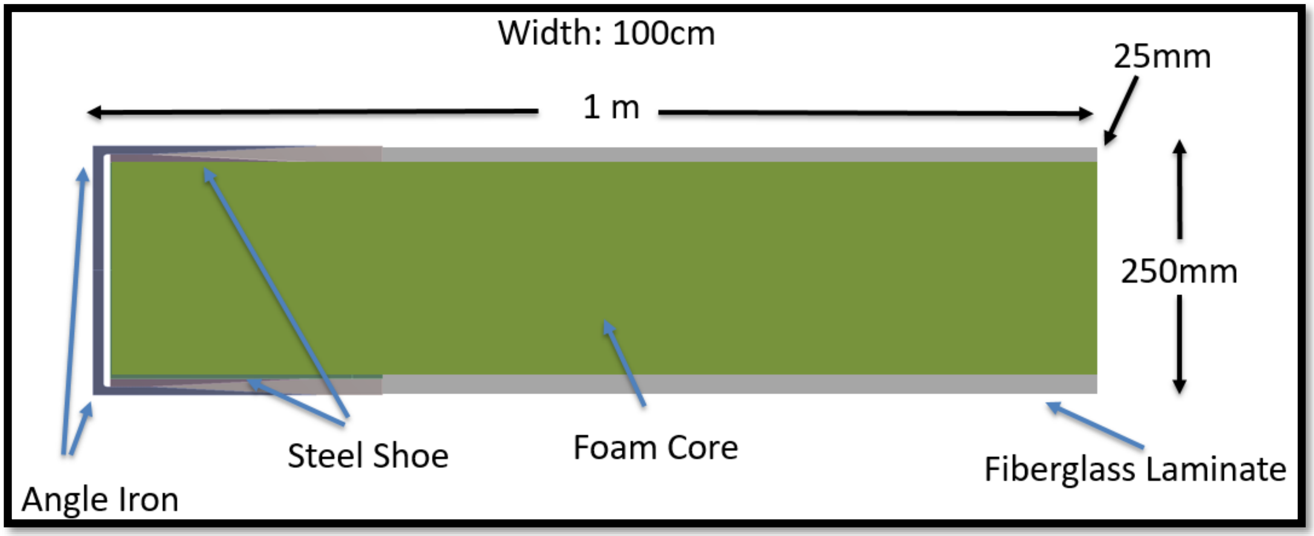

Figure 6. Beam Construction and Dimensions

There are many unanswered process questions regarding the construction of a sandwich plate with steel joints. Many of these questions will be answered at this time of prototype manufacturing. A detailed construction process will be followed and recorded such that a lessons learned can be applied to fabrication of the full-scale window.

8.2 BEAM LOADING

Beam Loading will be applied as cantilever and measured with strain gauges are multiple locations along the length of the beam. The load applied is a representative point load based on the hydrostatic load the entire plate will experience (estimated 3bar hydrostatic load). This loading will be compared to a sub-model analysis which will further validate the analytical model.

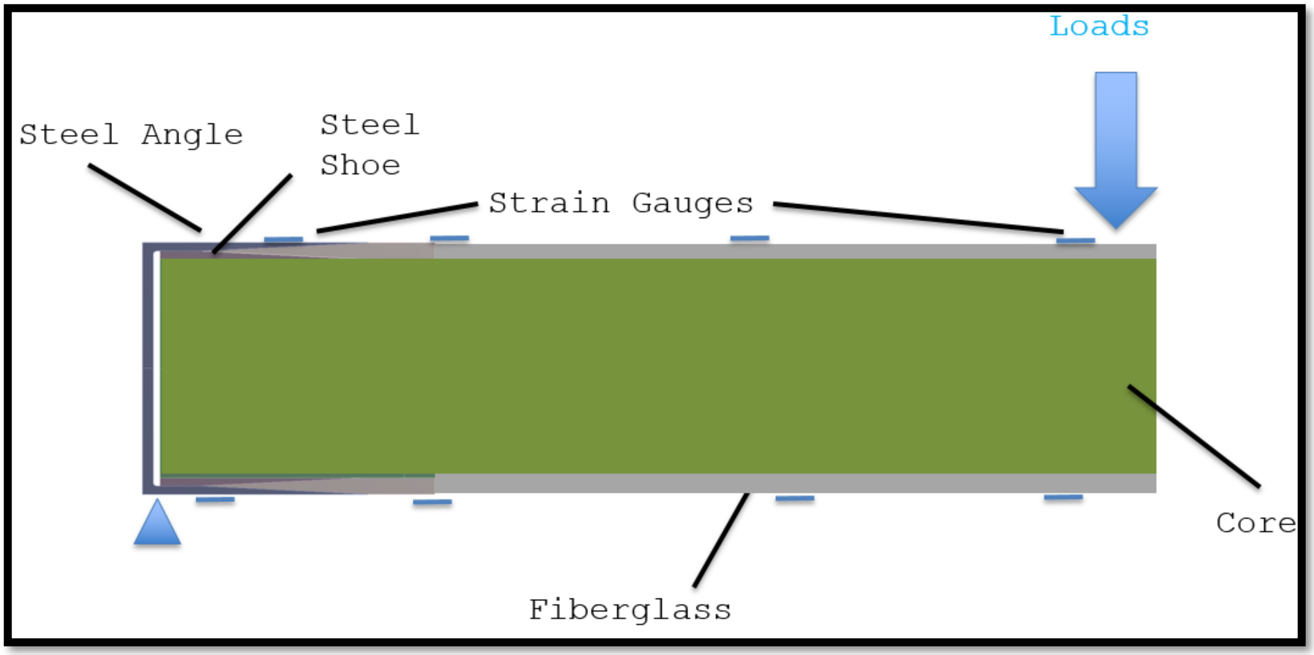

Figure 7. Beam Loading configuration

Load will also be applied as representative deflection at a value 1m from the edge of the plate within the analytical model. This measurement will also further validate the analytical model.

8.3 WELDING VALIDATION

In addition to material construction, there are concerns regarding integration between steel structures and composite materials. A majority of the detector is made from steel beams which will be connected to the steel framed composite window. This will likely be performed in some part with welding. Fiberglass, foam, and adhesives may not perform well near proximity of welding. A series of studies shall be performed to study a safe distance to weld from the fiberglass, foam core, and adhesive scarf joint locations.

8.4 COLD SUSCEPTIBILITY TEST

To address a concern of composite sandwich integrity when exposed to the cold temperatures of liquid argon within the cryostat a series of tests may be performed on a sandwich beam. This can be tested with two samples. The first control sample which is not subjected to any cold temperature. Then a second sample which would be submerged into LN2 temperatures prior to testing.

The tests to qualify integrity are based on a subset of *ASTM C481 Standard Test Method for Laboratory Aging of Sandwich Constructions*[3]

Shear Test	Test Method C273
Compressive Strength	Test Method C364 and C365/C365M
Delamination Strength	Test Method C363
Tensile Strength	Test Method C297/C297M
Flatwise Flexure	Test Method C393

Climbing Drum Peel Test Method D1781

8.5 FLAMABILITY

Common foam core materials are polyvinyl chloride (PVC), polyurethane (PU), polystyrene (PS), styrene acrylonitrile (SAN), polyetherimide (PEI) and polymethacrylimide (PMI).

May Halogen fire retardants be used? Core may need to be designed for fire resistance

Also consider the laminate properties for room temperature cured resins will begin to melt at 90°C

Appropriately address flammability concerns will require guidelines from safety and environmental committees.

9. WINDOW PLATE PROPERTIES

9.1 PRESSUREIZED LOADING (GAS TIGHT)

The final test to validate the composite window readiness level will be a subscale composite window. The size of the window is meant to be as large as possible without making manufacturing or testing significantly challenging. The scale chosen for this is 1/4

The window will undergo hydrostatic loading using pressurized air. Currently this is calculated using 350mBar of H20 and 4.5 meter tall Argon for a 1 bar load. With a safety of factor of 3, this is 3 bar or 43.5 psi. To perform this testing, a sealed steel box will be made that interfaces to the composite window and simulates the remaining structure of the cryostat. It will then be pressurized to 3bar. Stress/Strain on the window will be measured using strain gauges.

Additionally this test allows a measurement of leak rate. The composite window is ideally gas tight. That will be verified here by installing pressure gauges to the simulated structure.

This simulated build also allows development of Nondestructive Testing methods to ensure quality on the full-scale window build.

10. OTHER CONSIDERATIONS

10.1 Heat Susceptibility: Glass Transition Points and Heat Flexure Temperature

The glass transition temperature and heat flexure temperatures for resins are standard mechanical properties provided by manufacturers. These are not expected to be tested, but assumed to meet the manufacturer's material data sheet.

10.2 Handling Loads

The ¼ scale window will be useful for developing handling techniques and testing any handling loads. Such as standing the window up, does it need to be supported?

10.3 Electric Conductivity

Currently no electrical conductivity or faraday cage requirements are anticipated.

11. REFERENCES

[1] ASTM D3039 Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials
[2] D3410 Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading
[3] ASTM C481 Standard Test Method for Laboratory Aging of Sandwich Constructions
[4] Eurocode Basis of Structural Design EN 1990:2002