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Static friction testing of various materials in liquid nitrogen

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Abstract
The Deep Underground Neutrino Experiment will utilize charge readout planes (CRPs) that are submerged within liquid argon. These charge readout planes are part of a drift detector used measure the cascade of charged particles generated by the interaction of neutrinos with the argon nuclei. The CRPs require specially designed supports to ensure they remain precisely positioned even after the thermal contraction of the sensors and the cryostat that occurs during cooldown. These supports must also protect the cryostat floor and CRPs from potential damage due to thermal strain during cooldown. These objectives are satisfied by utilizing an intermediate slip plane within the support to provide a stress relief point with a lower friction coefficient than the interface between the sensor support and the cryostat floor. Therefore, the design of these supports requires knowledge of the static coefficient of friction for various materials at cryogenic temperatures. It was found that the coefficient of friction associated with materials such as metals, ceramics, and polymers have been studied very little at cryogenic temperatures. Specifically, there is not much data relative to the static coefficient of friction associated with polymers at low temperatures. Therefore, a test apparatus was developed to measure various materials' cryogenic static coefficient of friction with the geometry required for the support design. Several tests with different materials were carried out using this test apparatus to identify a material combination with very different static coefficients of friction that can be used for the membrane to support interface and intermediate slip plane.

1. Background/Relevance

The Deep Underground Neutrino Experiment (DUNE) is an international collaboration to study the nature of neutrinos. DUNE will utilize a particle detector module located at the Sanford Underground Research Facility in South Dakota. The particle detector is placed in a cryostat and has 160 individual Charge Readout Planes (CRPs) that are submerged in liquid argon and used to detect charged particle trails left from passing neutrinos. While filling the cryostat with liquid argon, there will be a significant thermal contraction of the cryostat membrane and the CRPs. Corrugations in the cryostat membrane ensure that the membrane has sufficient flexibility to prevent strain from building during this cooldown. However, the CRPs will contract significantly across their entire geometric length. To ensure that this thermal contraction does not damage the cryostat membrane, the CRPs cannot slide on the membrane floor during this process; however, it is also critical that the CRPs remain in a known position after thermally equilibrating with the liquid argon. This report describes the CRP supports designed to meet both requirements.

The CRP support design allows for a controlled and predictable final position of the CRP, which is important to minimize the data processing required during neutrino detection. Three of the four CRP supports have an intermediate slip plane that utilizes a material combination with significantly lower frictional resistance (coefficient of friction) than the materials associated with the support to membrane interface, shown in Figure 1. The fourth CRP support does not include the slip plane; therefore, this support will remain fixed to the membrane, and the other three will move during the cooldown process. The selection of materials used at the slip plane and the support to membrane interface is important and not obvious. The tribology depends on several factors, and the coefficient of friction at low temperatures is not well-studied. This paper describes the tests conducted using various materials to identify a material combination that creates significantly greater frictional resistance at the support to membrane interface than at the intermediate slip plane, thereby ensuring that slippage will not occur on the cryostat membrane while relieving stress in the CRP structure.

2. Support Design

Three important requirements constrain the design of the supports for the CRP. First, the support cannot damage the membrane floor during installation or while the CRP is thermally contracting. Second, the CRP must contract in a controlled, predictable manner. Third, the thermal contraction must not cause excessive forces that will damage the CRP itself. The support design developed to meet all these criteria has a support to membrane interface and an intermediate slip plane, as shown in Figure 1. The support includes a mechanism for centering the support while positioning the CRP during installation. This centering mechanism guarantees that the support has enough travel to account for all thermal contractions.

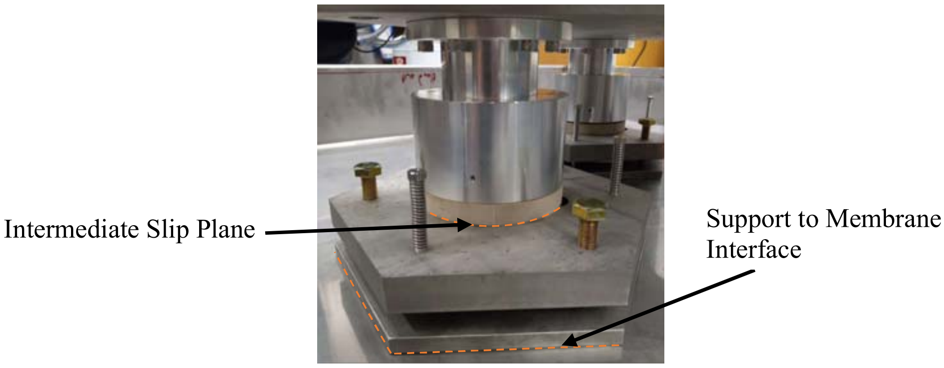

Figure 1: Special CRP support to ensure controlled positioning and controlled sliding during thermal expansion.

2.1. Intermediate slip plane design

The goal of the intermediate slip plane is to ensure that sliding motion does not occur on the membrane floor in order to prevent any damage to the membrane floor due to thermal contraction. To achieve this, materials with a lower coefficient of friction (COF) are used for the intermediate slip plane compared to those used in the support to membrane interface. The difference in COF between planes ensures that the friction forces on the membrane floor will be high enough that the metal footing will remain stationary (relative to the membrane) during the entire thermal contraction period. The membrane floor is made of stainless steel 316; therefore, all tests pertaining to the support to membrane interface were conducted with 16-gauge stainless steel 316 sheet metal.

The intermediate slip plane allows for controlled motion of the CRP during contraction. Each CRP has four supports, one of which will have a locked intermediate slip plane. The fixed slip plane support prevents any motion relative to the membrane. The combination of these supports allows the CRP to contract towards a singular focal point: the fixed foot. This support configuration results in predictable positioning of the CRPs after the thermal contraction is complete.

2.2. Centering mechanism

A mechanism was designed to center the footpad to the slip plane, illustrated in Figure 2, ensuring that the slip plane is centered when it is lowered but has enough travel during contraction to prevent binding. This design uses four springs and conical positioning posts to center the footpad when no load is applied to the support (i.e., during lowering of the CRP into position). Once a load, which depends on the stiffness of the springs installed, is applied to the foot, the spring will expand, allowing for free movement of the slip planes. The clearance associated with the design is 10 mm in any direction allowing for adequate travel from thermal contraction. The maximum estimation for thermal contraction expected is 7.5 mm. This value was calculated using the farthest possible position for the supports that can be mounted to the CRP, 3.626 m, and the coefficient of thermal expansion from the National Institute of Standards and Technology [1]. It was assumed that due to the design of the CRP structure, which uses plates and c-channels, the warp direction would be a good model for the nature of the CRP contraction.

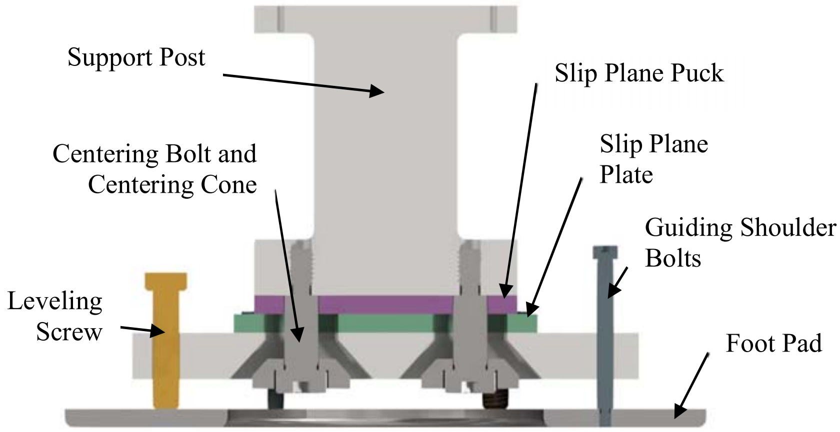

Figure 2: Cross section of support centering mechanism. Not illustrated are the springs which open the intermediate slip plane when support is unloaded.

2.3. Lower foot pad

The footpad in contact with the membrane floor can be leveled without disassembly. Leveling is achieved using three jacking screws, while springs are used to maintain proper assembly when unloaded. The ability for leveling and height adjustment is important to account for inconsistencies in the membrane floor.

3. Support Material Selection

To ensure that the CRP support functions properly, it is necessary to carefully consider the materials used in the slip planes. The coefficient of friction, strength, machinability, and coefficient of thermal contraction were all important while considering material selection.

3.1. Polymers

Once the CRP is installed, the support design keeps the intermediate slip plane under a constant compressive load. Therefore, the selected materials must not creep at room temperature and change geometry significantly before cooldown. To satisfy this requirement, when choosing polymers, only ones with a glass transition temperature above room temperature were considered for further testing.

Reduced temperature in polymers leads to changes in hardness, stiffness, and elastic modulus, affecting the cryogenic coefficient of friction. Friction data collected at room temperature may not reflect the behavior at 77 K. Also, tests carried out using dry surfaces may not reflect the coefficient of friction when submerged in liquid cryogen due to the presence of liquid cryogen [2]. In the DUNE project, the supports will be submerged in argon when most thermal contraction occurs. PEEK and nylon are used elsewhere on the CRPs and therefore have been approved for this application. It is important to ensure that the materials used do not negatively interfere with the overall scope of DUNE. Both polymers also fulfill before mentioned requirements as well. Therefore, they were considered as a potential material for the intermediate slip plane.

3.2. Metals

As the literature suggests, the friction coefficient of metals is generally temperature independent [3]. Using friction data at room temperature, different metals with a range of COF were selected, and then easy to machine and relatively affordable materials were prioritized. Of the materials, aluminum 6061 and stainless steel 316 were chosen for testing.

4. Test Equipment

The test equipment used to measure the friction coefficient is illustrated in Figure 3; it is comprised of a tub cryostat, linear motion device, test sled, load cell, and an enclosure.

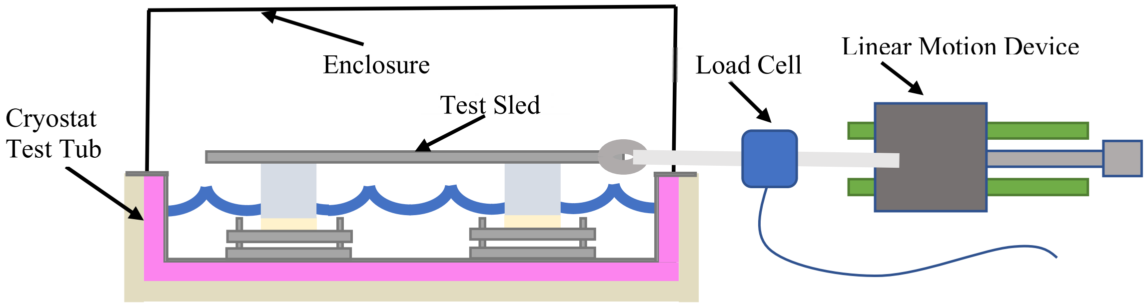

Figure 3: Schematic of test equipment.

4.1. Tub cryostat

The test enclosure is a tub cryostat, shown in Figure 4, made of 16-gauge 316 stainless steel insulated with polyethylene foam supported by a wooden structure. All seams are welded to make the tub liquid tight. The tub is designed to simulate DUNE's cryostat floor.

4.2. Test sled

The test sled is a solid plate to which the supports described in the previous section can be attached, and upon which reference weights can be stacked, see Figure 5. The sled has mounting locations on the front and back, allowing ropes to be attached for loading. The rope used for applying the load is made from nylon; nylon has sufficient elasticity to allow for a gradual increase in the force between the load cell and test sled during the displacement of the linear motion device alleviating the need for any additional compliance.

Figure 4: 316 stainless steel sheet metal Cryostat tube.

Figure 5: Test sled with reference weights before test.

Test pucks were made to assess different combinations of materials together. These pucks are fixed to a foot plate that can be fixed in place using stopper blocks. This design allows the frictional force required to cause slip to be recorded without requiring the fabrication of different posts and feet.

4.3. Linear motion device

A linear motion stage was used to apply a uniform and constant force. A ball screw moves a carriage linearly away from the test sled. The hand wheel turns the screw slowly to ensure a load reading can be identified and recorded during the slip. This device can be seen on the right side of Figure 6.

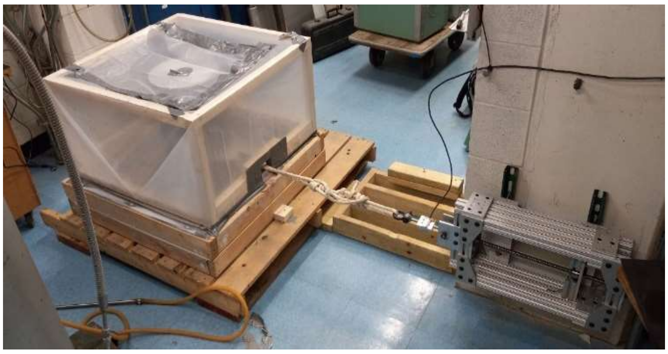

Figure 6: Entire test apparatus, including the enclosure.

4.4. Load cell
To record the reactionary force from the friction, a load cell, Rice Lake weighing systems RL20000st 500lb, is installed between the linear motion device and the test sled. The nylon rope was used to connect the linear motion device and the test sled to ensure that gradual force was applied to the test sled as the rope is made taught. To record data, a National Instruments NI 9237 was used to measure the load cell analog output signal and digitize it for processing in the LabVIEW program.

4.5. Enclosure
During initial tests, moisture from the air condensed and led to solid impurities in the liquid nitrogen (LN2) that affected the friction results. In the DUNE environment, air will have been purged from the cryostat prior to the cooldown. Therefore, an enclosure was built to prevent this ice formation, and tests were conducted to determine the importance of these impurities. A nitrogen blanket prevented atmospheric moisture from affecting the friction tests. The nitrogen blanket uses a wooden structure covered in plastic sheets. The enclosure is purged of air before filling with LN2 to ensure that no moisture is frozen anywhere, which would affect the coefficient of friction. The bottom seams are sealed with weather stripping and duct tape.

5. Test Process
Several tests were performed to ensure all material combinations would function properly during the cooldown. Two tests were designed to isolate the intermediate slip plane from the support to membrane interface. The first test uses stopper blocks between the tub and footpad, which isolated the intermediate slip plane by ensuring the footpad was fixed in the slip direction. The intermediate slip plane was locked to test the support to membrane interface, isolating the slip to the support to membrane interface. All tests were performed in a climate-controlled building at temperatures of approximately 295 K.

5.1. Calibration
A calibration test was performed to ensure that the test equipment was functioning properly. The calibration was done by hanging the known weight from the load cell to verify the force recorded by the data acquisition system. Loads from approximately 150 N to 2200 N were used and a deviation of approximately 1 N was measured through the entire range of loads.

5.2. Preparation of test equipment
Preparation steps were performed before cooldown to ensure the tests were repeatable. The supports were leveled with the jacking screw to provide consistent face pressure on the support to membrane interface. All mating surfaces that would be tested, membrane floor, support footing, and intermediate slip plane, were wiped with a clean, dry paper towel to clean and remove unwanted debris. Then all of these surfaces were cleaned with acetone to remove any oils or contaminants.

5.3. Selection of Liquid Nitrogen as test cryogen
DUNE will use liquid argon within the neutrino detector, yet for the friction tests, liquid nitrogen was used. The following factors justified this substitution: The University of Wisconsin-Madison facility has cheap and abundant access to liquid nitrogen. The viscosity of nitrogen and argon is 2.017E-07

m^2/s, and 1.880E-07 m^2/s, respectively. The density of nitrogen and argon is 807.7 kg/m^3, and 1397 kg/m^3, respectively. The boiling points of nitrogen and argon are 77 K and 87 K, respectively. Other groups within DUNE use liquid nitrogen as a substitute for liquid argon when testing components.

6. Test Results

It should be noted that these tests were meant to create a functional product for DUNE. This process resulted in a test matrix that was not complete. Testing for an individual material was concluded once a material was found to satisfy the requirements. Initial tests were repeated several times to ensure that the testing process was consistent. It is important to note that the geometry of the support significantly affects the value of COF [4].

6.1. Intermate slip plane tests

When calculating the COF for the intermediate slip plane, the weight of the support above the intermediate slip plane was used for the normal force calculation. It was initially thought that the dry room temperature tests would result in higher COF for PEEK compared to tests carried out submerged in LN2. However, our tests showed this not to be the case. These nonintuitive results resulted in a large number of additional tests carried out to verify that the results were not due to the test process.

After receiving nylon from the manufacturer, it was found that it had grooves on the surface related to the machining process. Surface imaging was performed to evaluate the magnitude of these machining grooves as seen in Figure 7. The nylon pucks were positioned so that the direction of motion would be parallel or perpendicular to these grooves.

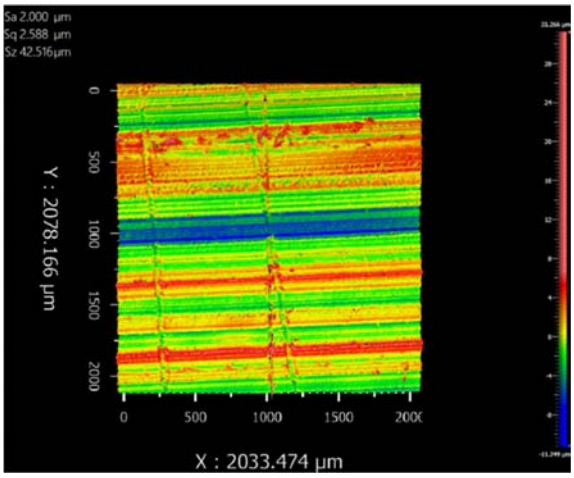

Figure 7: Surface roughness images of the machining grooves from a Zygo Newview 9000 3D optical surface profiler. Roughness values were as follows Sa 2.000 µm, Sq 2.588 µm, and Sz 42.516 µm.

The data from these tests were recorded in Table 1, and it should be noted that there is no significant difference in the mean COF observed between PEEK on PEEK and nylon on nylon 66. This realization resulted in eliminating nylon from consideration due to manufacturing imperfections.

Table 1: Test data from the isolation of the intermediate slip plane submerged in LN2 while enclosed. The = symbol represents the parallel test while the ⊥ represents perpendicular tests. The standard deviation was calculated by taking the square root of the sum of squared differences between each data point and the sample mean, dividing by the sample size minus one.

Material	Tests	Samples	Mean Static COF	Standard Deviation
PEEK on PEEK	3	83	0.27	0.017
Nylon 66 on nylon 66 =	2	21	0.26	0.054
Nylon 66 on Nylon 66 ⊥	2	21	0.35	0.182

6.2. Support to membrane interface test

When calculating the COF for the support to membrane interface, the weight of the entire support was used to calculate the normal force. All combinations of polymers tested resulted in a negligible difference in frictional resistance compared to the stainless steel on stainless steel result. The small difference in COF resulted in an effort to identify materials that provide a substantially larger frictional force for the support to membrane interface. Tests were conducted with aluminum foot pads on stainless steel membrane that resulted in significantly higher frictional forces than those found for the intermediate slip plane. Data from the support to membrane interface tests can be seen in Table 2.

Table 2: Test data from the isolation of the support to membrane interface while submerged in LN2.

Material	Tests	Samples	Mean COF	Static	Standard Deviation
Aluminum on Stainless Steel	7	97	0.34		0.017
Stainless Steel on Stainless Steel	3	97	0.20		0.006

6.3. Impact of Moisture

During the initial testing, it was observed that there was a significant amount of moisture which condensed and froze onto the test sled surface which is illustrated in Figure 8. Due to concerns about additional friction forces that might be caused by this moisture, an enclosure was designed and built to remove moisture from the LN2 testing environment, see Figure 6. Dry gaseous nitrogen is blown into the enclosure for at least 15 minutes to provide a nitrogen blanket to the system. Once the cryostat tub was filled with LN2, the boiling provided a positive pressure in the enclosure to prevent atmospheric air from entering the system.

Figure 8:Test without enclosure resulting in ice buildup.

A grinding crunching sound was noticed during tests that had clear signs of moisture buildup in the LN2. These tests also resulted in significantly higher friction forces, as seen in Table 3. The presence of impurities in the cryogen significantly affects the friction forces. Due to the unpredictable and uncontrollable nature of these impurities, it is advised that little to no moisture is present in the cryostat while cooldown occurs for optimal performance of the supports.

Table 3: The static COF comparing the differences with and without an enclosure.

Material combination	Mean (SD) without encloser	Mean (SD) with encloser
Aluminum on Stainless Steel	0.44 (0.010)	0.34 (0.017)
Stainless Steel on Stainless Steel	0.23 (0.033)	0.20 (0.006)

6.4. Polymer Testing

During the testing of the polymers, the results recorded during 77 K tests were unexpectedly high compared to the room temperature tests. It is known that polymers change hardness with decreased temperature [5]. It is also known that friction is a function of hardness for metals [6]. The room temperature tests were also performed in a dry environment whereas the 77 K test submerged the slip plane in LN2. The frictional forces recorded are static, and the lubrication properties of LN2 may not be as significant as in a dynamic system, but, as currently understood, the COF of a polymer should not be greater for at low temperature case. Table 4 shows that the measured COF for PEEK on PEEK did tend to increase substantially at cryogenic temperature.

Table 4: A comparison of room temperature tests to 77K for PEEK on PEEK.

	Temp	Test	Sample	Mean COF (SD)
PEEK on PEEK	295 K	3	45	0.216 (0.023)
	77 K	3	83	0.274 (0.017)

7. Conclusion

There were two significant observations associated with the tests described in this paper. First, moisture present in the cryogenic tests significantly affects the frictional forces. The presence of moisture increasing COF is intuitive, but it is believed that there could be similar effects associated with moisture retained within the polymer that causes an increase in friction when submerged within LN2. To prove this theory, more tests would have to be performed by controlling the moisture level in the polymer before testing. To fully understand the nature of this system, more tests are required. Tests at room temperature would be ideal to help better understand the effects of temperature on the polymer's frictional properties. Retesting PEEK on stainless steel with an enclosure would also be helpful to compare to the other tests.

Second, there was no significant difference between the tested polymers and stainless steel. This slight deviation of COF led us to conclude that the best solution for our support would be to use stainless steel as the intermediate slip plane. Using stainless steel should provide a sufficiently low COF and a predictable result. The cost of materials is not significantly different between PEEK and stainless steel. Iteration to the support design is in process, and further testing will be performed to verify this conclusion.

8. References

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