A test rig for analysis of adhesive tapes at 4 K cryogenic temperature

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Abstract. Cryostats and dewar vessels, in particular those used for liquid helium applications, are usually equipped with multi-layer insulation (MLI). Thereby, multiple foils are wrapped around the respective vessels, tubing and components. As standard, different foils are bonded edge to edge using adhesive tapes either based on aluminized non-metallic films or on aluminum foil. There are a number of standard test procedures for adhesive tapes near ambient temperatures (e.g. AFERA 5012/ISO 29863) allowing a standardized characterization of tapes in terms of holding force and long-term reliability. Unfortunately this does not hold true for adhesive tapes to be used at cryogenic temperatures. In this respect, a test rig comprised of a spring-based traction mechanism has been developed by the authors. Combined with a liquid helium dewar, the fabricated test set-up allows a precise and reproducible application of an adjustable tensile load at 4.2 K and measurements of the respective holding time. In the following, the overall set-up including its significant features is described and first experimental results with aluminum tapes are presented.

1. Introduction

In cryogenic industry adhesive tapes are widely used for various applications. For instance aluminum tape is applied on the outer side of a steel vessel (cold mass) inside the vacuum space to improve the radiative properties of the steel surface. This means that the adhesive of the tape is directly applied on a cold surface (i.e. 4.2 K for a LHe vessel or 77 K for a LN₂ vessel). Detached tape might cause a thermal link e.g. between a cooling shield and the vessel which holds the cryogenic liquid. One possible use of double sided tape is to fix a multi-layer insulation (MLI) blanket to the bottom of a vessel to prevent sagging in the middle. Here the same very low temperature environment applies and a potential failure of the tape is very likely to degrade the insulation performance.

Of course most of the tapes used at low temperatures were initially not designed for this application. Furthermore changes of the carrier material using the same adhesive may change its performance at cryogenic temperature due to differential thermal contractions. Since the cryogenic industry is a relatively small market (compared to automotive and other big series production industries), the effect of certain changes to a tape and its adhesive on its strength at cryogenic temperatures may not merit the same attention by the manufacturer as other properties.

At the moment there is no established standard to qualify the tape for low temperature applications. Using a test method established for room temperature in cryogenics is difficult. A mechanical 180° peel test at room temperature of a 125 mm long tape shows a more or less steady peel force over the
full length [1]. In contrast to that at cryogenic temperatures an instantaneous failure at a certain load is expected and the material disconnects almost simultaneously over the whole adhesive area.

Additionally the influence of the bending stiffness of the tape carrier is much bigger than at room temperature. Tests with mechanical test rigs at very low temperatures also showed that the shock loads, i.e. a minor overshoot of the applied force during start-up, disconnect the tape from the substrate. This is intrinsic to all displacement controlled machines.

A different method is described in the AFERA 5012/ISO 29863 standard, where the time to failure with constant load is measured [2]. Here the measurement of the time to failure of several identical samples in a common test rig is suggested. The mean time to failure is used to compare different tapes. To apply this standard in a cryogenic environment a new test rig comprised of a spring-based traction mechanism has been developed and will be presented in the following chapters.

2. Design and Setup

The test method and the apparatus developed in the presented work are widely based on the AFERA 5012/ISO 29863 procedure A. The design is adapted for the use inside a standard 100 liter liquid helium dewar. The left sketch of Figure 1 shows the schematic design of the developed test rig. The insert with the tape sample is immersed into a LHe bath through the neck tube of the LHe dewar. Inside of a 12 mm by 0.5 mm stainless steel tube a tie rod is led from the cryogenic insert up to room temperature. By means of a spring force meter the mechanical load is applied outside the vessel at room temperature. The spring force meter is held by a spindle support, which allows an exact manual adjustment. A butterfly nut is used for linear adjustment of the spindle.

The centre of Figure 1 shows a magnified view of cold parts of the equipment. It consists of a moveable carriage sledge and a fixed counter block. The actual test surface of 12 by 12 mm is located on the carriage, which slides smoothly by means of a guide rail and is pulled by a tie rod. The counter block of identical width is fixed to the guide rail.

![Figure 1. Design of the developed test rig for adhesive tape testing inside a LHe dewar; Left: overall arrangement; Center: cold end with test surface; Right: slide rail with carriage](image)

To ensure maximum parallelism between the two designated surfaces, both components were manufactured out of one single aluminum block (ENAW-6082). The material was milled to the desired depth and surface roughness and then cut into two parts. Considering thermal shrinkage at low temperature both carriage and guide rail were made of aluminum. A tight tolerance of the sliding block to the guide rail prevents any significant tilt angle between test surface and the counter surface.
The support structure at ambient temperature is mounted on top of the liquid helium dewar. The spring force meter is fastened to an adjustable spindle at the upper side and at the lower end it is linked to the tie rod (see Figure 2). The tie rod is centred by a brass bushing on top of the 12 mm by 0.5 mm stainless steel tube. Shear stress is applied by means of tension through the spring load. A time measurement and a contact switch are installed to monitor the time to failure of the tape sample. Test rig and dewar vessel are placed on a scale to control the liquid filling level of the dewar. Through the entire test the probe is completely immersed in the liquid phase of the respective cryogenic liquid.

3. Test Method

Based on AFERA 5012/ISO 29863 standard (procedure A) a new test method has been developed, retaining parts of the surface preparation procedure, the preparation of test specimen and conduction of experiments. At first, all test surfaces were thoroughly cleaned using isopropyl alcohol to remove residual adhesive and other contaminations. In a following step acetone was used to remove any remaining residuals. After drying the surface, a 70 mm long stripe of the test specimen was cut to the desired width (12 mm, respectively 6 mm). In order to apply the test specimen to the dedicated surfaces, a 5 mm diameter rubber roll was used, loaded with a 1 kg mass. By that a constant and reproducible contact pressure was ensured during tape application.

In the next step, the test section was carefully lowered into the cryogenic bath and cooled down. Then the spring load was carefully applied and slowly raised. The load was continuously monitored by the operator.

**Figure 2.** Liquid helium dewar vessel with mounted test rig and components at ambient temperature visible on top. The spring force meter is held by a spindle support. For linear actuation a butterfly nut is installed at the top end for manual adjustment of the spring elongation.
4. Measurements

4.1. Preliminary tests
The test rig was designed to ensure free motion of the aluminum carriage at room temperature and at cryogenic temperatures. This was verified by testing the bare apparatus under various conditions. A basic load was measured at room temperature and when cooled by a liquid nitrogen bath, 0.5 N and 1 N for the two test apparatus. These values comprise the gravimetric force of the tie mechanism, the aluminum carriage and the dynamic friction forces. The static friction is not quantifiable, respectively below the resolution of the spring force meter.

3M-425 Aluminium Tape was used for reference measurements and for better understanding of the new test rig. The tape is made of a 0.08 mm thin annealed aluminum liner with acrylic adhesive [3]. In order to ease these tests a liquid nitrogen bath was used instead of liquid helium at first. By additional tests in liquid helium it was ensured that identical parameters and results show up at this elevated temperature.

At all cryogenic temperatures a rather high bonding strength was observed. At 77 K the 3M-425 Aluminum Tape resisted shear loads up to 49 N for an extensive holding time (more than 2 days). The maximum applicable force was limited to 49 N by the installed spring scale. Thus an increase of the load was not easily feasible. The results are summarized in Table 1. Thus, in order to extend the range of specific shear stress, the test area was reduced from 12 mm by 12 mm to 6 mm by 12 mm.

Table 1. Results of preliminary tests using 3M-425 Cryotape at room temperature and in a LN$_2$ bath.

| Test area | Load [N] | Cooling method | Time to failure | Observations       |
|-----------|----------|----------------|-----------------|-------------------|
| 1 12 x 12 mm | 14.7     | Room temperature | 2-3 min         | Slippage and fail* after 2-3 min |
| 2 12 x 12 mm | 49       | LN$_2$         | -              | No failure for 2 days |
| 3 12 x 12 mm | 49       | LN$_2$         | -              | No failure for 2 days |

* adhesive failed

4.2. Cleaning method
A series of experiments were dedicated to figure out a proper cleaning method. The reduced test area of 6 mm by 12 mm was used. The AFERA standard suggests solvents like Acetone, Methanol or Methyl ethyl ketone for cleaning. Here a combination of Isopropanol and Acetone was used. A set of 6 tests at room temperature did not show any dependency on the sequence of cleaning procedures. The tape failed within a few minutes in all cases at about 7.4 N (Table 2). Hence the cleaning procedure was reduced to clean once with Acetone.

The contamination with a fingerprint, meaning touching the previously cleaned test area with the thumb and thereby contaminating the surface, did not change the shear strength. The tape seems not to be very sensitive to any residuals at the test area. Table 2 shows the test results with different cleaning methods.

4.3. Measurements in LN$_2$ and LHe
Finally a series of measurements was conducted in a liquid nitrogen bath and in a liquid helium bath. The previously described cleaning method (1 x Acetone) was applied and a test area of 6 mm by 12 mm was used. The apparatus was immersed in the cryogenic bath without any load. After sufficiently long cooling time the load was gradually increased until the maximum force was reached or the tape failed.
Table 2. Test results for three different cleaning methods to prepare the test area and resulting observations.

| No. | Cleaning method | Load [N] | Cooling method | Time to failure | Result |
|-----|-----------------|----------|----------------|-----------------|--------|
| 1   | 3 x Acetone 1 x Isopropanol | 9.81 | Room temperature | 0 | Fail* during increasing load |
| 2   | 3 x Acetone 1 x Isopropanol | 7.4 | Room temperature | 0 | Fail* during increasing load |
| 3   | 1 x Acetone | 7.4 | Room temperature | 3 min | Fail* |
| 4   | 1 x Acetone | 7.4 | Room temperature | 30 s | Fail* |
| 5   | 1 x Acetone + fingerprint | 7.4 | Room temperature | 3 min | Without fail |
| 6   | 1 x Acetone + fingerprint | 7.8 | Room temperature | 30 s | Fail* |

adhesive failed

Even at the reduced area of 6 mm by 12 mm no failure at the maximum load of 49 N of the 3M-425 Aluminium Tape was observed. Two long term tests inside a liquid helium bath were conducted. After 482 h (approx. 20 days) and 571 h (approx. 23.8 days) at 49 N no failure was observed and the test was stopped. No slippage or plastic strain of the adhesive tape is observed at all at cryogenic temperatures. The time to failure measurement according to the AFERA standard is not applicable, since the maximum adhesive strength of the tape exceeds the expected range by more than a factor of five. All test results are shown in Table 3.

A typical course of action on the part of typical cryotape users is to precut the tape and “park” it aside, i.e. temporarily sticking it to an adjacent surface. Then it is removed and attached to its designated point of application. Tests number 3 and 4 were intended to investigate this. It turned out that the adhesive strength of the tape was still out of range and no failure was observed, i.e. no degradation was found.

Table 3. Test results for 3M-425 Aluminum Tape at LN₂ and LHe temperature.

| Test area | Load [N] | Cooling method | Time to failure | Observations |
|-----------|----------|----------------|-----------------|--------------|
| 1 6 mm x 12 mm | 49.05 | LN₂ | n/a | No failure |
| 2 6 mm x 12 mm | 49.05 | LN₂ | n/a | No failure |
| 3 6 mm x 12 mm (“parked”) | 44.15 | LN₂ | n/a | No failure |
| 4 6 mm x 12 mm (“parked”) | 44.15 | LHe | n/a | No failure for 50 h |
| 5 6 mm x 12 mm | 44.15 | LHe | n/a | No failure for 6 h |
| 6 6 mm x 12 mm | 49.05 | LHe | n/a | No failure |
| 7 6 mm x 12 mm | 49.05 | LHe | n/a | No failure for 482 h |
| 7 6 mm x 12 mm | 49.05 | LHe | n/a | No failure for 571 h |
5. Findings
The analysis of adhesive tapes at cryogenic temperatures showed surprising results. The 3M-425 Aluminium Tape exceeds the AFERA standard by far. No failure was observed even with five times higher load and only 50% of the required test area.

In summary the findings of described measurements up to now are:

a) At cryogenic temperatures adhesive tapes can achieve very high holding force. Much higher than at room temperature.

b) No slippage or creep of the tape neither at LN$_2$ nor at LHe temperatures was observed.

c) The AFERA standard is not applicable.

6. Future work
It is planned to continue the cryogenic tape tests. Higher mechanical loads are desired in order to determine the adhesive strength of the 3M-425 Aluminum tape and other test specimen.

References
[1] ISO 8510-2:2006. Adhesives –Peel test for a flexible-bonded-to-rigid test specimen assembly
[2] AFERA Test Methods Manual, 2008 edition. 5012 (ISO 29863-EN1943). Test method for Static Shear Adhesion of Pressure Sensitive Tape
[3] 3M 425 Aluminum Foil Tape. Product data sheet. [www.3m.com](http://www.3m.com) (07/19/2016)