

# **IMPROVEMENT STUDY ON UNLINED FILAMENT-WOUND COMPOSITE TANK DESIGNED FOR CRYOGENIC PROPELLANTS**

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## **ABSTRACT**

Feasibility investigations of CFRP composite as applied to unlined CFRP cryogenic propellant tank for reusable vehicle systems have been conducted by National Space Development Agency of Japan (NASDA). Reflecting the results of the first two phases of pressurization test on small prototype filament-wound (FW) tank with 300mm diameter conducted from room temperature to liquid nitrogen temperature, the improved third phase of FW tank were conducted and the result was reported in this paper. As a part of the investigations, elementary assessments on the temperature-dependence of CFRP shear performance and strength performance of the promising adhesive material EA9394 were simultaneously executed from room temperature to cryogenic temperatures and the results were also reported here.

## **1. INTRODUCTION**

National Space Development Agency of Japan (NASDA) has been conducting the feasibility investigations [1~5] of CFRP composite as applied to unlined CFRP cryogenic propellant tank for reusable vehicle systems. Two phases of pressurization tests on small prototype filament-wound (FW) tanks with 300mm diameter were conducted from room temperature to liquid nitrogen temperature in our previous studies. The specific CFRP material chosen was called Q-133 CFRP prepreg: which is concluded high-toughness and middle-elasticity epoxy resin #133 reinforced by BESFIGHT IM600.

The first phase test of FW tank was conducted in 1999. The tank was a 300mm long cylinder with 300mm-diameter hemisphere domes at both ends. The flanges on both sides were made of SUS304 stainless steel and sealed by rubber O-rings. 10mm-width tape prepreg was used to wind on a plaster mandrel and cured in an autoclave at 180°C for 2 hours. Leakage was happened during waterproof test and the tank was cut into pieces for analyzing the weak points. The second phase test was conducted from 2001 to the beginning of 2002 and the second FW tank was manufactured with improved design and manufacturing processes. The image configuration of the improved design for second phase FW

tank is shown in Fig 1. The Q-133 prepreg tape was changed into 3.5mm width reflecting the investigation results of the first phase FW tank. Nickel alloy flange was used to match the thermal expansion with Q-133 CFRP. There were some large wrinkles concentrated on half surface of the completed tank (we called it unhealthy area) and the other half surface looked better which was called healthy area with some small wrinkles. Leakages were occurred at 0.3MPa pressurization test along a large wrinkle in the unhealthy area. After repairing the leakages with EA9394 adhesive, waterproof tests at room temperature with 1.0MPa and 1.5MPa internal pressures were followed on the repaired tank. After that cryogenic pressurization tests were followed with 1.5MPa and 1.9MPa internal pressures without any leakages happened in the healthy area. The first leakage in the healthy area was happened with 2.0MPa internal pressure at cryogenic temperature. Based on the detailed inspection of the cross-sectional tank wall, it concluded that the leakages were connected with the scale of wrinkles. The shrinkage of plaster mandrel during curing process and the tension control during winding were considered as the most two possible reasons for the occurrence of wrinkles and they have to be confirmed.

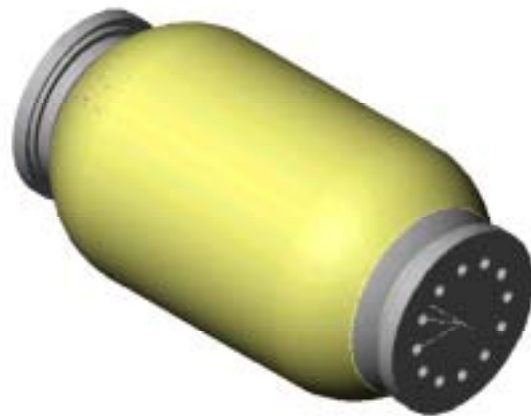


Fig 1 Image Configuration of Second Phase FW tank

Based on the results reflection of the above-mentioned first two phases of pressurization tests on 300mm-diameter prototype filament-wound (FW) tanks, the third phase FW tank was manufactured to confirm that if the shrinkage of plaster mandrel during curing process would lead up to the leakages. Two-step curing process was introduced to avoid the plaster mandrel shrinkage and the tension control was kept the same with the second phase tests. It concluded that changing curing process couldn't avoid the wrinkle occurrences and the leakages were happened all over the tank surfaces during the snoop tests.

As a part of the investigations, elementary assessments on the temperature-dependence of CFRP shear performance and strength performance of the promising adhesive material EA9394 were simultaneously conducted from room temperature to cryogenic temperatures and the results were also reported here. From CFRP shear testing, the results demonstrated that shear strength of in-plane shear specimens almost doesn't change with temperature, while for inter-laminar shear the shear strength becomes higher as the temperature decreases. From EA9394 adhesive testing, we confirmed that as the temperature cooled down to cryogenics the tensile strength of EA9394 increased while the shear strength decreased.

## 2. THRID PHASE OF FW TANK WITH DIFFERENT CURE PROCESSING

The third phase of FW tank test was planned with the reflection of the results of the second phase tests. Here we are interested in confirming the mandrel shrinkage caused by cure processing as the main reason for wrinkle occurrences.

### 2.1 Improvement for FW tank manufacturing

Based on the investigation results obtained from the second phase test on the 300mm-diameter prototype filament-wound (FW) tank, the third phase FW tank was manufactured to confirm that if the shrinkage of plaster mandrel during cure processing would lead up to the leakages. Instead of curing the FW tank at 180°C about 2 hours, two-step cure processing was introduced trying to avoid the shrinkage of plastic mandrel during the cure processing. The detail condition of two-step cure processing was 5 hours at 140°C plus 2 hours at 180°C, which was decided after the confirmation on mandrel itself and the hardening results of CFRP prepreg. The winding angle was also changed from 30° to 37° to meet the mandrel shape design requirement. The winding pattern was  $[37^\circ_2/90^\circ_2/37^\circ_2/90^\circ_2/37^\circ_2]$  just as same as in the second phase test. The tension control during the winding was also kept the same with the second phase tests: for both helical and hoop winding the same tensile force was designed for tank manufacturing.



Fig 2 The Third FW Tank after Cure Processing

### 2.2 Penetration test

Figure 2 shows the photograph of third FW tank after curing process. Wrinkles were observed over the tank surface. From this point we confirmed that the wrinkles of the FW tank was not caused by the shrinkage of plastic mandrel. To assess the damages from manufacturing process, penetration test using fluorescence was firstly executed after the FW tank was completed. Fig 3 shows one sample of the tank surface near wrinkles under fluorescence searchlight. From this test some damages were observed along the wrinkles.

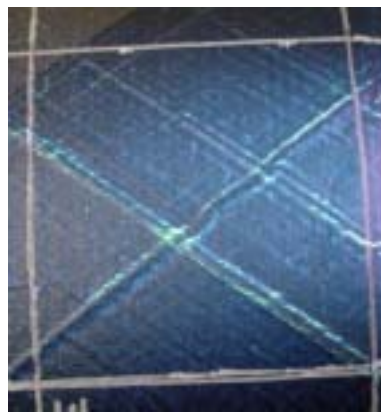


Fig 3 Penetration Test Result on Tank Surface

### 2.3 Waterproof test at 1.0MPa

0.4MPa and 1.0MPa waterproof test was then conducted followed the penetration test. Leakages were

observed over the tank surface as shown in Fig 4a. To identify the leak points, we conducted snoop test and found all the leak points as shown in Fig 4b. From this phase tank, leakages from the hoop winding part at the flange necks were observed for the first time. Another different point with the second phase tank was the serious leakages occurred at the interfacial area between doom and cylinder.



(a) Waterproof Test



(b) Snoop Test

Fig 4 Waterproof Test and Snoop Test of FW Tank

## **2.4 Discussions**

In this phase test, two-step cure processing for CFRP tank was introduced to prevent the shrink of plastic mandrel. From this point we can confirm that the shrinkage of the plastic mandrel has nothing to do with the occurrences of wrinkles over the FW tank surface. Another considerable reason for the wrinkles left was the tension control during filament winding. Because leakages from the hoop winding part at the flange necks and from the interfacial area between doom and cylinder were observed for the first time, more detail inspection of the cross-sectional tank wall need to be executed after cutting the tank to pieces.

## **3. ELEMENTARY STUDIES OF STRENGTH CHARACTERIZATIONS**

### ***3-1 Shear performance evaluation of CFRP laminates***

In developing low-weight CFRP (carbon fiber reinforced plastics) liquid propellant tank systems, the cryogenic shear performance of CFRP becomes very important for ensuring safety and durability because liquid propellants. Usually the plastic matrix component dominates the shear performance of CFRP. Mechanical behavior of the plastic matrices is very sensitive to temperature, especially the

cryogenic temperatures at which the molecular motion comes as close as theoretically possible to ceasing completely. The shear performance of CFRP laminates is thus very sensitive to temperature changes. There have so far been only a few studies on the shear performance of CFRP at very low temperatures, especially at cryogenic temperatures.

Here we are interested in the shear behavior of Q-133 CFRP material over temperatures ranging from room temperature to cryogenic temperatures (lower than  $-150^{\circ}\text{C}$ ), especially at cryogenic temperatures. The doubly v-notched specimen shown in Fig 5 was introduced for experimental study.

The specimen size was followed the ASTM standard [6] fitting the modified Wyoming test fixture. The radius of the notch roots is fixed at 1.3 mm and the thickness of specimen is fixed at 2.2mm following the ASTM standard.

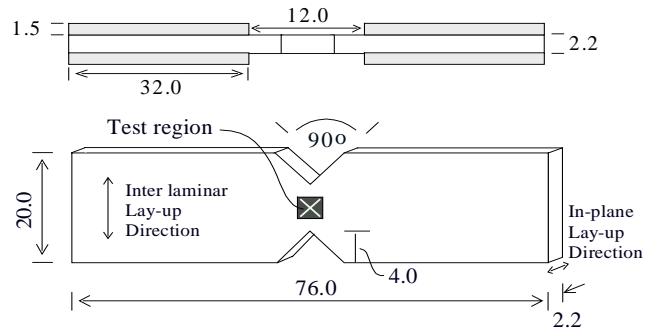


Fig 5 V-notched Specimen for Shear Testing (mm)

CFRP laminates with total 16-cross-ply ( $0^{\circ}/90^{\circ}$ ) structure for in-plane and total 160-cross-ply ( $0^{\circ}/90^{\circ}$ ) structure for inter-laminar shear tests in different lay-up directions were first fabricated using unidirectional Q-C133 CFRP prepreg sheet of 0.14mm thickness per layer. Vacuum-bagging pressurization was executed for every four plies about three minutes to minimize the void contents. The v-notched specimens were then cut from the CFRP laminates based on the shape and size as shown in Fig 5 using a diamond-cutting machine. Aluminum tabs (1.5mm thickness) were introduced following the ASTM recommendation and bonded to CFRP v-notched specimens with EA9394 adhesive having high bond strength at very low temperatures. Fig 6 presents photographs of both in-plane and inter-laminar specimens with different lay-up directions near the notch root areas. The stacked  $\pm 45^{\circ}$  strain gauge rosettes (Micro-measurements KFG-1-120-D16-11N-15C2; also shown in Fig 6) are introduced with 1.0 mm gauge length covering an area of about  $0.5 \text{ mm}^2$  at the specimen center for strain measurements. Testing was conducted at three temperatures: room temperature,  $-70^{\circ}\text{C}$  and cryogenic temperature at  $-160^{\circ}\text{C}$ .



(a) In-plane Shear



(b) Inter-laminar Shear



(c) Strain Gauge

Fig 6 Photo Images of V-notched Specimens and Strain Gauge Used

Fig 7 shows the rupture shear strengths obtained from in-plane and inter-laminar shear testing at different temperatures. These rupture shear strengths are calculated from the failure loads divided by the cross-sectional area between two notch tips. These results demonstrate that the failure shear strength of in-plane shear specimens almost doesn't change with temperature, while for inter-laminar shear the failure shear strength becomes higher as the temperature decreases to cryogenic temperature. This result is encouraging because the delamination of CFRP laminates generally becomes more severe with temperature changes.

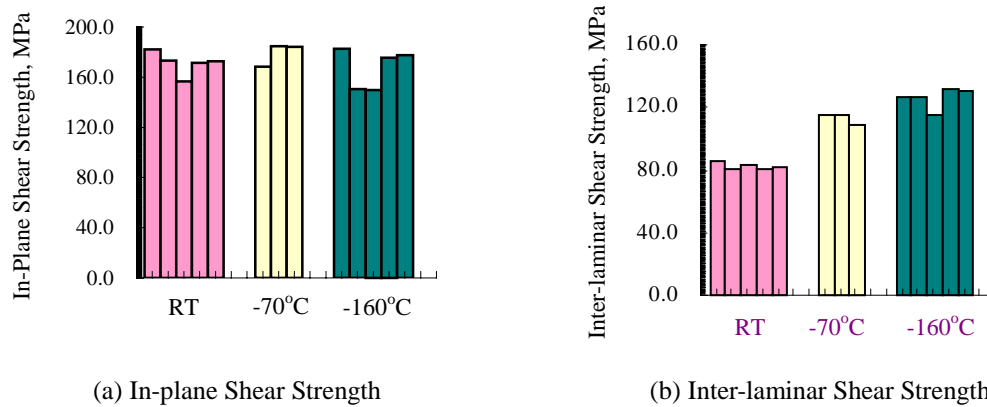
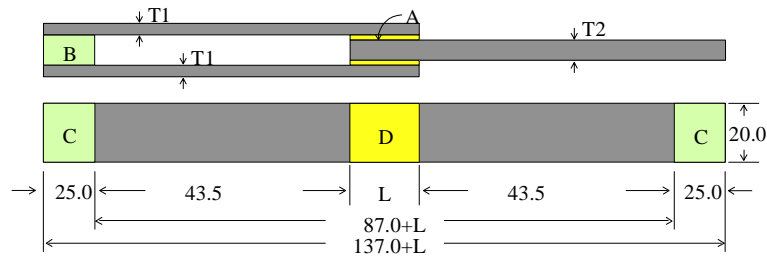


Fig 7 Rupture Shear Strength Obtained from V-notch Shear Testing

### 3-2 Tensile and shear strengths of bulk adhesive EA9394

In the development of CFRP liquid-propellant tank system for Reusable Launch Vehicle (RLV), it's also important to clarify the material properties of adhesive bonds at cryogenic conditions. The bond strength of three types of adhesive materials (FM300, HT435 and EA9394) as applied to two different CFRP adherends (IM7-UD and T300-UD) were experimentally evaluated at room temperature and two cryogenic temperatures (-160°C and liquid helium (LHe) temperature at -269°C) through double-lap tensile specimens in our previous studies. It concluded that HT435 and EA9394 adhesives have stable and relatively higher cryogenic performance of bond strength for both CFRP adherends studied. Here we are interested in the cryogenic performance of bulk EA9394 adhesive, a two-part structural paste adhesive cured at room temperature, to understand the failure mechanism of adhesive bonds at cryogenic conditions. Tensile and shear strength of bulk EA9394 adhesive were measured from room temperature to cryogenic temperature based on ASTM standards.

**Shear Strength Testing:** At first the shear strengths of bulk EA9394 adhesive were evaluated with four types of bond thickness (0.1mm, 0.2mm, 0.5mm and 1.0mm) based on ASTM standard D3528-96 (Double-lap tensile test) [7] from room temperature to cryogenic temperatures. The double-lap tensile specimen with shape and size as shown in Fig 8 are introduced following ASTM standard and also designed to fit the cryostat for liquid helium testing. Aluminum (AL/5020) is used as adherend material following the recommendation of ASTM standard.



T1: 1.0 mm; T2: 2.0 mm; L: 12.7 mm

A: Test Glue Lines ; B: Spacer ; C: Area in Test Grips; D: Shear Area

Fig 8 Double-lap Specimen Followed the ASTM Standard

Fig 9 shows the failure loads obtained from above-mentioned EA9394 double-lap tensile test at different temperatures. First of all one can see that the shear strength of EA9394 adhesive decreased with the bond thickness increased. This might be considered because of the quality inferior caused by the thicker bond. Secondly from these results we can see that the cryogenic shear strength of EA9394 adhesive almost doesn't change so much compared with the room temperature results, and for thicker bond the cryogenic shear strength becomes higher.

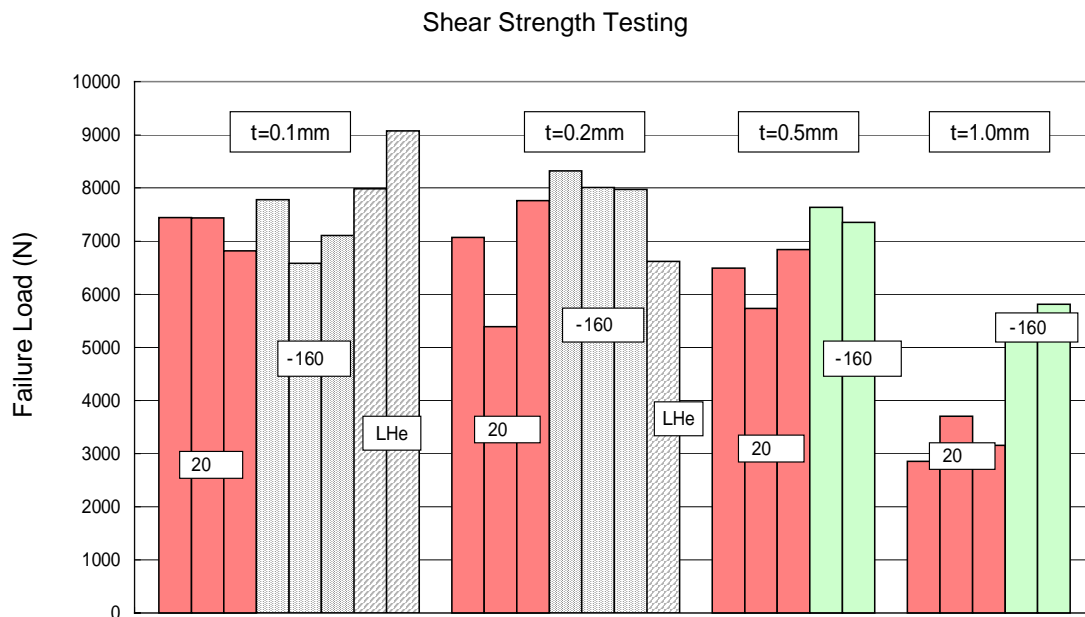


Fig 9 Shear Strength of Bulk EA9394 Adhesive at Different Temperatures

**Tensile Strength Testing:** Tensile strength of bulk EA9394 adhesive was also evaluated with two types of thickness (0.5mm and 1.0mm) based on ASTM standard D882-01 [8] from room temperature to cryogenic temperatures. The tensile specimen with shape and size is shown in Fig 10 following ASTM standard and also designed to fit the cryostat for liquid helium testing.



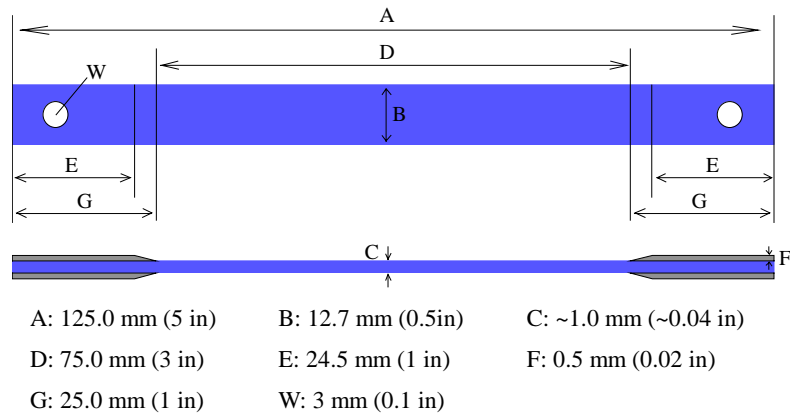


Fig 10 Tensile Specimen with Thickness Less Than 1.0mm Followed the ASTM Standard

Fig 11 shows the tensile strength obtained from above-mentioned tensile tests at different temperatures. From these results one can see that the tensile strength of EA9394 adhesive decreased with the bond thickness increased. This might also be considered because of the quality inferior caused by the thicker specimen manufacturing just like the case of shear strength testing. Secondly we can also see that for same thickness the cryogenic tensile strength of EA9394 adhesive becomes higher by comparing with the room temperature results.

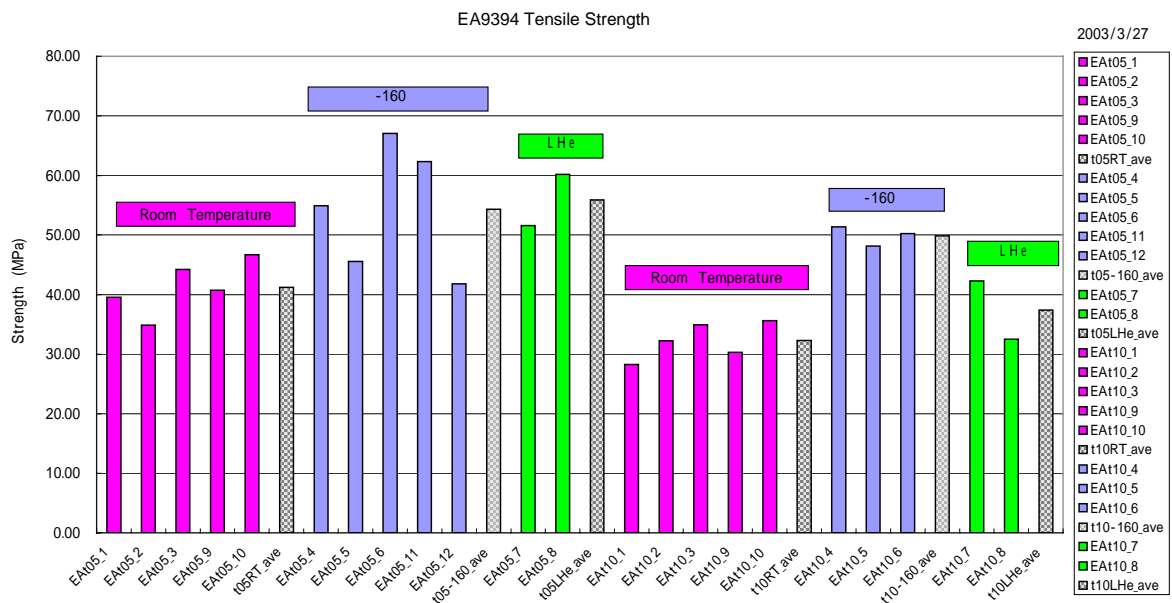


Fig 7 Tensile Strength of Bulk EA9394 Adhesive at Different Temperatures

## CONCLUSIONS

- The shrinkage of plastic mandrel during cure processing is confirmed having nothing to do with the wrinkles occurred on the FW tank surface. Leakages from the hoop winding part at the flange



necks and from the interfacial area between dome and cylinder were observed for the first time, more detail inspection of the cross-sectional tank wall need to be executed after cutting the tank to pieces.

- Failure shear strength of in-plane shear specimens almost doesn't change with temperature, while for inter-laminar shear the failure shear strength becomes higher as the temperature decreases to cryogenic temperature.
- Cryogenic shear strength of EA9394 adhesive almost doesn't change so much compared with the room temperature results, and for thicker bond the cryogenic shear strength becomes higher.
- Cryogenic tensile strength of EA9394 adhesive becomes higher by comparing with the room temperature results.

## REFERENCES

1. Morimoto, T., Shimoda, T., and Morino, Y., "Cryogenic Test of CFRP Scale Tank for the Future Rocket Plane Propellant," Proceedings of the 37<sup>th</sup> Aircraft Symposium, pp. 541-544, Tokyo, November 1999
2. S. Cantoni, G. Di Vita, G. Totaro, Morino, Y., and Morimoto, T., "Applicability of Filament Winding Method to Fabricate Cryogenic Composite Tanks for Liquid Propulsion," IAF-00-S.3.08
3. S. Cantoni, G. Di Vita, G. Totaro, Morino, Y., Morimoto, T., and Shimoda, T., "Cryogenic Composite Tanks for Liquid Propulsion" 2<sup>nd</sup> International Symposium Atmospheric Re-entry Vehicles and Systems, 2001, Arcachon
4. Shimoda, T., Morino, Y., Ishikawa, T., Morimoto, T. and S. Cantoni, "Study of CFRP Application to Cryogenic Fuel Tank for RLV," 7th Japan International SAMPE Symposium, Tokyo, November 2001
5. Shimoda, T., He, J., Morino, Y., Morimoto, T., "Study of CFRP Application to Cryogenic Fuel Tank for RLV," 23rd International Symposium on Space Technology and Science, Matsue, Japan, May 2002
6. Standard Test Method for Shear Properties of Composite Materials by the V-notched Beam Method, ASTM Standard D5379-93, American Society for Testing and Materials, Philadelphia, May, 1993
7. Standard Test Method for Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading, ASTM Standard D3528-96, American Society for Testing and Materials, Philadelphia, Sept., 1996
8. Standard Test Method for Tensile Properties of Thin Plastic Sheeting, ASTM Standard D882-01, American Society for Testing and Materials, Philadelphia, Oct., 2001