The Application of Carbon Fiber Composites in Cryotank

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Abstract

To meet the design goal for lightweighting the next-generation launch vehicles, carbon fiber reinforced polymeric-based composites are being explored for cryogenic fuel tank applications. The applications of carbon fiber composites in liquid hydrogen (LH2) and liquid oxygen (LOX) fuel tanks were introduced in this chapter. The materials, processing, and design of DC-XA LH2 tank, X-33 LH2 tank, SLI LH2 tank, and CCTD Program tank were discussed. Lockheed Martin LOX tank and Space X LOX tank were introduced. Technology development, materials development, and development trend of cryogenic fuel tanks were discussed. Thin-ply hybrid laminates and out-of-autoclave tanks are projected for future space missions.

Keywords: carbon fiber reinforced composites, LH2 tank, LOX tank, hydrogen permeability, cryogenic properties

1. Introduction

Carbon fiber reinforced resin matrix composite materials (CFRC) are being used in the aerospace industry as a means of reducing vehicle weight. CFRC have advantages in high strength-to-weight and high stiffness-to-weight ratios. For future heavy lift launch vehicles and space exploration structures, advanced lightweight composites will be fully utilized in order to minimize vehicle weight, and CFRC in space applications requires rigorous development to demonstrate robustness, durability, and high factors of safety.





Figure 1. Delta IV heavy.

The future heavy lift launch vehicles require extremely high propellant mass fractions to achieve the designed performance. This drives the designers to incorporate lightweight materials in as many structures as possible. Propellant fuel tanks account for a large proportion in the launch vehicles, both of structural mass and geometric space. Approximately 60% of the dry mass of a launch vehicle is the fuel and oxidizer tanks. The implementation of composite cryogenic propellant fuel tanks (cryotank) for future heavy lift launch vehicles could greatly reduce the vehicle's weight by replacing the identically sized cryotanks constructed of metallic materials. United States' Committee on Materials Needs and R&D Strategy for Future Military Aerospace Propulsion Systems reported that composites offer the potential for the greatest mass reduction of all of the materials for tank [1–3].

For the case of Delta IV heavy lift launch vehicle, as shown in **Figure 1**, compared to Li-Al fuel tank, the weight saving of upperstage composite cryotanks were 43 and 26%, respectively [1].

In addition, composite design could reduce fabrication cost. Delta II faring, Delta III faring, and interstage production data have shown that composite launch vehicle structures are less expensive than metal ones [2].

Graphite-epoxy composite cryogenic tank development began at Boeing (then McDonnell Douglas) in 1987 and continues today, primarily for reusable launch vehicles (RLV) and heavy lift vehicles.

2. LH2 cryotank

The cryogenic tanks are the dominating components of the vehicle structure. To achieve weight reduction of the next-generation launch vehicles, carbon fiber reinforced polymericbased composites are being explored for cryogenic liquid fuel tank. A composite cryotank structure can save 30% by weight than lithium aluminum alloy.

Cryogenic composite tank development began in 1987. First, most of the effort has been devoted to liquid hydrogen tanks mainly because liquid hydrogen tanks are larger than liquid

oxygen tanks, lightweight materials would provide a proportionately greater weight reduction. The other reason was a common assumption that organic composite materials could not be used in LOX tanks [4].

NASA has been exploring advanced composite materials and processes to reduce the overall cost and weight of liquid hydrogen (LH2) cryotanks while maintaining the reliability of existing designs.

Composite liquid hydrogen tank development in NASA went through the National Aerospace Plane (NASP) program, the Single-Stage-to-Orbit (SSTO) vehicle X-33 program, and the recent second generation RLV applications under the Space Launch Initiative (SLI) and the Composite Cryotank Technology Demonstration Project (CCTD) (2011–2014) [5]. The fundamental issues for composite liquid hydrogen tanks are the hydrogen permeability and cryogenic mechanical properties of the composite material.

2.1. DC-XA composite LH2 cryotank

Liquid hydrogen is an essential but highly volatile fuel used in launch vehicles. It must be stored and used at -253°C, a temperature that causes most materials to become quite brittle. Liquid hydrogen also has an extremely fine molecular structure, which allows it to seep through the tiniest gaps. Therefore, liquid hydrogen tanks present an extreme challenge in engineering materials because of hydrogen permeability and cryogenic properties [2, 3].

Work in composite cryogenic tank development at McDonnell Douglas (MDA) began in 1987. MDA solved the problem of hydrogen permeation and cryogenic properties of composites successfully, and the composite LH2 tank for DC-XA was designed, developed, and fabricated. The tank was 8 feet (2.43 m) in diameter, 16 feet long (4.88 m) [6], constructed with IM7/8552 toughened epoxy material from Hercules. Automated fiber placement (AFP) was employed



Figure 2. DC-XA composite LH2 cryotank.

to manufacture the cryotank. The tank was incorporated with lightweight internal insulation modeled after the Saturn S-IVB design. The composite tank was 33% lighter than the DC-X tank.

DC-XA provided the first flight tests in real-world operating environments for composite liquid hydrogen tank in 1996, as shown in **Figure 2**. The tank passed the ground and flight tests successfully. It experienced approximately 55 pressure cycles throughout the ground and flight test program, demonstrated the acceptability of composite liquid hydrogen tank for future launch vehicle [7, 8].

2.2. X-33 composite LH2 cryotank

Lockheed Martin Space Systems Company (LM) is one of the world leaders in large cryogenic tank technology [9]. The composite LH2 cryogenic tank for X-33 vehicle was designed, developed, and built by LM and test by NASA. The tank was a conformal, load-bearing, composite sandwich structure, consisting of an outer facesheet, honeycomb core, and inner facesheet as shown in **Figure 3a**. The inner and outer facesheets were constructed with IM7/977-2 and the core was constructed with KorexTM+3-pcf honeycomb. **Figure 3b** shows the overall dimensions of the tank. The tank is 28.5 feet in length, 20.0 feet in width, and 14.0 feet in height [10, 11].

The sandwich LH2 tank of the X-33 Demonstrator (see **Figure 4**) was ground-tested at Marshall Space Flight Center on November 3, 1999. It failed this validation test when the outer skin and core of the sandwich separated from the inner skin, although the inner skin was undamaged, several fractures were observed in the outer skin in the same lobe.

After extensive study, NASA determined that the delaminations were likely caused by a combination of factors. The most probable cause of the failure was determined to be a combination of the following phenomena [1, 12–14]:

- Microcracking of the inner facesheet with gaseous hydrogen (GH2) infiltration
- Cryopumping of the exterior nitrogen (N2) purge gas

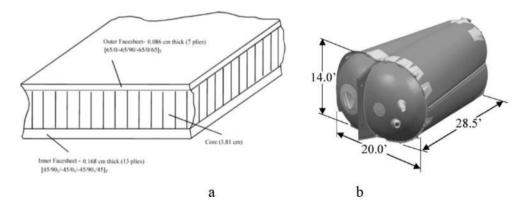


Figure 3. Lockheed X-33 composite LH2 cryotank (a) configuration of honeycomb sandwich structure and (b) tank dimensions.

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Figure 4. X-33 vehicle.

- Reduced bondline strength and toughness
- Manufacturing flaws and defects
- Infiltration of GH, into the core, which produced higher than expected core pressures.

This setback did not discourage NASA to further develop composite cryotanks. The X-33 project was the first of its kind in large-scale composite cryotanks. The tank design was very novel with a number of cutting edge technologies. The delamination failure during the testing provided a valuable case study for NASA and Lockhed Martin to continue the composite material application in cryotank.

2.3. Space Launch Initiative (SLI) Composite Cryotank Program

Northrop Grumman designed, developed, and fabricated a cylindrical composite LH2 cryotank for Space Launch Initiative Composite Cryotank Program, as shown in **Figure 5**. The tank was constructed as a sandwich structure with carbon fiber composite skins sandwiched

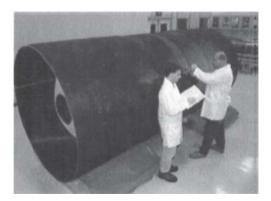


Figure 5. North Grumman's LH2 cryotank [15].

between nonmetallic honeycomb core. By using thinner carbon fiber laminars and increased number of cross laminates, the microcracks in the composite skins were significantly reduced by a factor of 16.

Other major innovations in this program included the multifunctional sandwich core and the out-of-autoclave process. Taking the advantage of the sandwich structure, hydrogen permeation was stopped by a barrier of aluminum foil between the inner skin and the honeycomb core. Any hydrogen leakage was vacuumed out through the perforated honeycomb core. In addition, the core acted as a thermal insulation layer. The cryotank manufacturing avoided the expensive autoclave by the novel ultrasonic tape lamination approach. The ultrasonic energy provided excellent compaction while depositing the carbon fiber tapes. The final composite structure was cured in an oven at ambient pressure.

The testing involved the simulated load cases close to a rocket launch. The composite cryotank, in 1.8 m diameter and 4.5 m long, was filled with liquid hydrogen. The tank was also subject to an internal pressure of 827 KPa and an axial load along the vertical axis of a launch vehicle. The composite tank was ¼ scaled size of a typical reusable launch vehicle cryotank. Further tests were performed to fill, apply internal and external loads, and drain the tank repeatedly 40 times, in order to study its structural integrity under cryogenic temperatures and its reusability [14–16].

The Northrop Grumman/NASA team proved that a cryogenic fuel tank made from composite materials has the structural integrity to withstand the mechanical and thermal stresses associated with repeated fueling and simulated launch cycles.

2.4. CCTD composite cryotank

The Composite Cryotank Technology Demonstration Project (CCTD) was part of the Space Technology Program and the Game Changing Development (GCD) Program for NASA. [17]. The program had a clear focus to deliver the proven technologies that would be deployed in future space flight demonstration. An extensive full-scale ground-based testing and lab experimentations were implemented. The development and demonstrations outlined in the CCDT Program were based on the relevant aerospace industrial experience over the last 20 years. The project successfully designed, manufactured, and tested a full-composite LH2 cryotank [18, 19]. The composite cryotank reportedly achieved 30% saving in weight and 25% in cost, compared with a baseline aluminum alloy cryotank.

NASA worked with four competing industry partners—ATK, Boeing, Lockheed Martin, and Northrop Grumman at the design phase of the project with the aim to reduce weight by 30%, reduce cost by 25%, and withstand a strain of 0.005 [20]. Four competing conceptual designs differing in materials, structures, manufacturing processes were assessed through coupon testing and finite element stress analysis.

A sample of the design requirements is shown in **Figure 6**: an inner tank that could contain pressurized LH2 at cryogenic temperatures, fitted with an outer skirt that could take the axial compression loads during launch-vehicle takeoff. All of the phase I concepts were required to use Government furnished information (GFI) IM7/977-2 lamina material property.

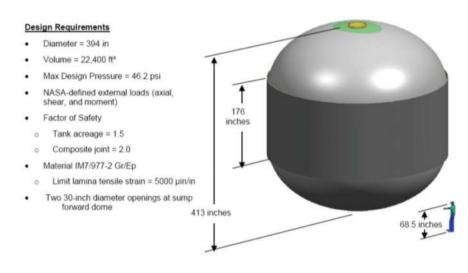


Figure 6. NASA reference Cryotank geometry and high level requirements [1].

The NASA team developed a metallic aluminum alloy cryotank concept for comparison to three industry IM7/977-2 composite concepts with the same overall dimensions: Boeing fluted core, Lockheed-Martin externally stiffened, and Northrop Grumman sandwich, as shown in **Figure 7**. The weight comparison for the four concepts is shown in **Table 1**. All three composite concepts exceeded the 30% weight reductions also had designs where the laminates were at or under the 5000 $\mu\epsilon$ strain limit desired by the CCTD Project when compared to the metallic cryotank. All of the three tanks were fabricated by automated fiber placement process. A cost analysis effort showed that 20–25% cost saving can be achieved by utilizing AFP process.

The results further showed that thin plies (65 or 70 g/m^2) are effective in resisting microcracks and thereby minimizing LH2 permeation.

Boeing's design and analyses showed that when designing to a 5000 $\mu\epsilon$ limit strain level, a 39% weight saving over a comparable aluminum-lithium tank designed using mature materials and manufacturing techniques can be realized. A cost analysis effort showed that 20–25% cost saving can be achieved by utilizing an automated fiber placement process [20].

Phase II of CCTD involved the design, analysis, fabrication, and testing of large scale (2.4-m diameter precursor and 5.5-m diameter demonstrator) composite cryotanks. The two tanks incorporated the design features and strain levels that represent a full-scale (8.4-m diameter) Space Launch System (SLS) propellant tank. Design features included a one-piece wall design that minimized tank weight.

Both tanks were fabricated at Boeing using automated fiber placement on breakdown tooling. The tanks were made of Cytec's CYCOM 5320-1 out-of-autoclave (OOA) prepreg, hybrid laminate was employed, using a combination of thick plies (145 g/m²), which can be placed relatively quickly and enable large (up to 10-m diameter) cryotank fabrication, and thin plies (70 g/m²), which create a microcrack-resistant laminate that helps prevent hydrogen permeation [21, 22] (**Figure 8**).

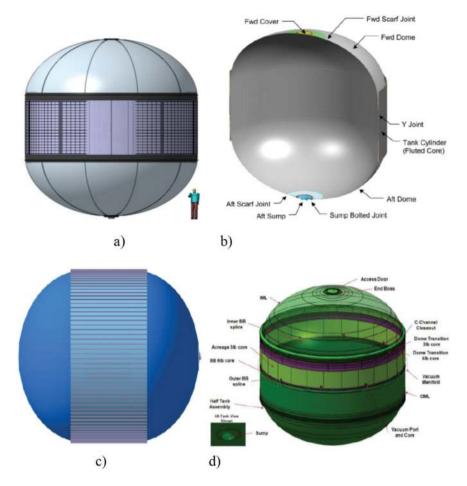


Figure 7. Four Cryotank concepts [1, 19](a) NASA al-Li concept, (b) Boeing fluted core sandwich Wall concept, (c) the Lockheed-Martin externally stiffened concept, and (d) Northrop Grumman composite honeycomb sandwich concept.

Company structural concept	Weight/lb	Weight saving/%
NASA TRL9 metallic baseline	10,925	_
Boeing fluted core	6696	38.7
Lockheed External Box Stiffened	6572	39.8
NGC Honeycomb Sandwich	6252	42.8

Table 1. Comparison of the three industry composite designs to the metallic design from NASA.

The 2.4-m diameter all-composite precursor tank was fitted with a laminate skirt that could take the axial compression loads during launch-vehicle takeoff and tested on June 25, 2013 at NASA MSFC, the tank was successfully pressure tested. The test met all requirements: stepwise fill



Figure 8. 2.4-m diameter tank, a robotic arm applies composite laminate [21, 22].



Figure 9. 5.5-m diameter tank [21, 22].

with liquid hydrogen (-223°C) to 90% volume capacity followed by pressurizing the tank to 931 KPa. The 2.4 m tank was then cycled through 20 pressure/vent cycles, measuring hydrogen gas permeation on the tank dome [21] (**Figure 9**).

NASA Space Launch System (SLS) has an 8.4-m diameter (8.4 m) Ares Vupper stage. NASA chose a 5.5-m diameter test article for the second phase of the program. Boeing was the only partner to produce and test this size tank with the existing infrastructure. A 5.5-m diameter cryotank was of sufficient scale. To prevent the delaminations occurred in X-33 composite cryotanks, subscale 5.5-m diameter CCTD demonstrator tank was fitted with an innovative fluted-core skirt.

NASA completed a demanding series of tests inside the test stand at MSFC. On March 26, 2014, the 5.5 m cryotank was subjected to flight loads in combination with pressure loads. Structural loads were applied to simulate the stress during a space flight. Liquid hydrogen at 253°C was filled in the composite tank. At the same time, a cyclic pressure of 138 to 207 KPa was applied to the tank.

The 5.5 m tank was pressurized to 400 KPa, reaching a maximum acreage strain of 5136 microstrain and demonstrating safety factors above 1.5 in the scarf joints. At the 5136 test strain, the permeation performance does not meet the CCTD goals, but well within the allowable for an upper stage or boost stage composite tank application [21].

The hydrogen permeation measured in the CCTD tanks are likely due to the porosity, estimated to be approximately 3%, from the low-pressure curing having facilitated void and crack formation, even in the thin plies.

The project confirmed that composite cryotanks can achieve a 33% weight savings compared to aluminum-lithium cryotanks, and it demonstrated permeation performance that meets the allowable for upper stage and boost stage applications.

This is the first effort to successfully build and test a tank of 5.5-m diameter [21, 22].

There are several accomplishments in CCTD cryotank of phase II:

(1) 5320-1 OoA epoxy resin matrix

Boeing tested a new material-Cycom 5320-1 that does not require expensive autoclave curing. Cycom 5320-1 is a toughened epoxy prepreg resin system designed for out-of-autoclave manufacturing of primary structures. With a lower curing temperature, the resin system is suitable for prototyping where low cost tooling or vacuum-bag-only curing is required.

Cycom 5320-1 handles similarly to standard prepreg. The difference is that the vacuumbag-only cured composites produce the quality equivalent to the autoclave process, with minimum porosity and competitive mechanical properties. It was received as thin sheets of B-stage film [23, 30].

(2) Thin ply prepreg-70 g/m², hybrid laminate

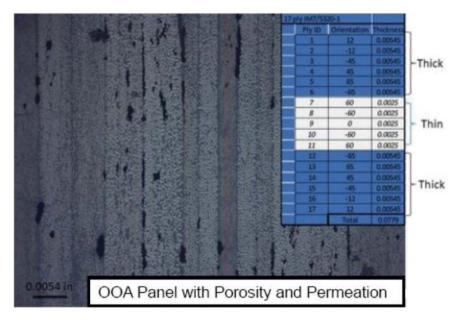


Figure 10. Thin and thick ply.

To reduce hydrogen permeation levels, hybrid laminate combination of thick plies (145 g/m²), which can be placed relatively quickly and thin plies (70 g/m²), which create a microcrack-resistant laminate that helps prevent hydrogen permeation was used in CCTD tanks.

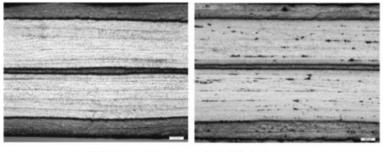
For further evaluation, hybrid laminates fiber-placed panels were produced at Boeing and evaluated at MFSC. The stacking sequence of the laminate was showed in **Figure 10**. The laminates were made up of 12 plies of 5.4 mil and 5 plies of 2.5 mil material. The report noted that when thin plies are used and standard laminate consolidation is achieved, permeation performance requirements will be met with very large margins.

Thin-ply composite structures offer many advantages in composite tank manufacture. They are far more resistant to the formation of microcracks. Also, tougher resins have been developed that offer protection against microcracks (may be used in conjunction with the thin plies). The down-side of the thin plies is that they make manufacturing more difficult. Present development efforts are exploring the use of fiber placement equipment to place the thin plies. Hybrid laminates are demonstrating the same performance as the thin plies. Excellent permeability results are achieved by both methods [25].

(3) Out-of-autoclave cure processing



Figure 11. Out-of-autoclave processing.



Autoclave cure

Out-of-Autoclave cure

Figure 12. Comparison of autoclave and out-of-autoclave processing.

Autoclave process is the major cost in composite manufacturing. The investment of the large size autoclave oven is formidable for any company. Boeing employed OOA resin matrix, and the tank exhibited approximately 3% porosity (**Figures 11** and **12**).

For further evaluation of OOA processing, two-hybrid laminates fiber-placed panels produced at Boeing and LH2 tested at MSFC. The laminates were made up of 12 plies of 0.137 mm and 5 plies of 0.064 mm material. The OOA laminates exhibited approximately 4% porosity.



Figure 13. Fluted-core skirt.



Figure 14. Four different composite tanks of CHATT project.

Testing with autoclave coupons and the same materials did not show measurable permeability. Evaluation of the OOA laminates revealed that microcrack forms in the thin plies, primarily due to the porosity in the laminate. To deduce porosity, and eliminate permeability for OOA, it is necessary to increase the number of thin plies and to reduce porosity by improving the OOA materials architecture and fiber placement processes.

(4) Robotic AFP

Instead of the gantry-based automated fiber placement (AFP) equipment classically used to build large cylindrical parts, Boeing opted to use robotic fiber placement (AFP). Robotic AFP system enabled improved capabilities-better reach in the dome areas and lower shaft clearance.

(5) Fluted-core composite skirt

To prevent the delaminations occurred in X-33 composite cryotanks, Boeing opted an innovative fluted-core skirt, comprising large trapezoidal members-technically, laminate-angled web members with structural-radius fillers between facesheets [24]. The fluted-core skirt was designed to take the compressive load during launch and to vent permeated hydrogen. The skirt structure was co-cured to eliminate potential de-bonding issues at the working temperature of 253°C (**Figure 13**).

2.5. CHATT project

The project Cryogenic Hypersonic Advanced Tank Technologies (CHATT) is a part of the European Commission's Seventh Framework Programme and run on behalf of the Commission by DLR-SART in a multinational collaboration. The main objective was to investigate carbon fiber reinforced composite material for cryogenic fuel tank applications (**Figure 14**).

Four different subscale CFRP-tanks have been designed, manufactured, and tested. The CHATT project contributed to significant progress in the design of composite tanks for cryogenic propellant applications in Europe. The European Technology Readiness Level (TRL) of such cryotank is in the range between three and four, while the TRL in the US is considerably more advanced [25].

3. LOX cryotank

The weight reduction provided by composite Lox tank is too great to disregard. The most difficult issue for composite Lox tanks is material compatibility. After solving the problems of the hydrogen permeation and cryogenic properties of LH2 tank, MDA and NASA investigated the compatibility of composite materials and liquid oxygen (LOX). The test results proved that composites could be used to fabricate Lox tanks for launch vehicles, and the results sufficiently convincing to plan on building and flying composite Lox tanks in X-33 and X-34 vehicles [26].

NASA and Lockheed Martin jointly developed the first composite cryogen tank for liquid oxygen storage. Lockheed Martin designed and manufactured a sub-scale LOX cryotank, while NASA tested it at its MFSC facility.

The composite tank is approximately 2.7 m in length and 1.2 m in diameter, and weighs less than 225 kg, the weight saving is 18% compared to metal tank of similar construction. The



Composite LOx tank Composite LOx tank testing

Figure 15. Composite lox tank of LM.



Figure 16. The 12-m diameter composite lox tank of space X.

tank passed the initial testing of cyclic liquid oxygen loading. The tank withstood the thermal and pressure conditions similar to that on a space launch vehicle. The tank did not permeate nor crack after 52 cycles of fill-drain liquid oxygen test, and passed the demonstrated test, as shown in **Figure 15** [27].

This tank marks a real advance in space technology. No approved standards for composite pressure vessels exist; there has not been enough information on them to write standards. So the technical data getting from this effort is very valuable [24].

Space X has designed, developed, and fabricated a composite LOx tank, which is a key component of interplanetary transport system (ITS). The tank is 12 m in diameter, and passed 2/3 exploration pressure test successfully in Nov 2016, see **Figure 16**. The carbon fiber used in the tank was provided by Toray. The 12-m diameter tank was the largest vessel ever produced [27].

4. Materials

Fiber-reinforced composites can be optimized and tailored with the right amount of fibers based on the directions and magnitudes of the stress state. Composite cryotanks can be the most efficient isotensoid structure. Yet the challenges remain with carbon fibers particularly for manufacturing large-scale cryotank.

IM7 which is manufactured by Hexcel, was used widely in cryogenic tank in NASA. IM600 which is made by Toho, was used in cryotank in Japan [28]. T800H which is made in Toray, its property parameters are equivalent to IM7 and IM600 (**Table 2**). All of them are intermediate modulus fibers, high tensile strength, PAN-based fiber, their properties are basically equivalent.

The carbon fiber with intermediate modulus, high tensile strength was used widely in composite cryotank manufacturing. The higher the modulus of carbon fiber, the higher the crystallinity of the fiber, this would lead to reduced surface active functional groups and decreased the interlaminar shear strength of composites. Low modulus carbon fiber is not good for

	Fiber type	Tensile strength/ MPa	Tensile modulus/ GPa	Strain/%	Density/g/cm ³	Filament diameter/µm
Toho	IM600	5790	285	2.0	1.80	5.0
Hercules	IM7	5300	275	1.8	1.77	5.2
Toray	T800H	5490	294	1.9	1.81	5.0

Table 2. Property parameters of carbon fibers.

	IM7/5320-1 [31]	IM7/977-2 [32-34]	IM7/8552 [34]
0°Tensile strength/MPa	2703	2690	2650
0°Tensile modulus/GPa	156	165	168
Poisson's ratio	0.34	_	_
90°Tensile strength/MPa	81	75	_
90°Tensile modulus/GPa	9.7	7.6	_
0°Compressive strength/MPa	1737	1580	1690
0°Compressive modulus/GPa	143	152	150
Short beam shear strength/MPa	119	112	128
CAI/MPa	176	262	234
Open-hole tensile strength/MPa	498	448	_
Open-hole compressive strength/MPa	386	310	_

Table 3. Properties parameters of IM7/5320-1and IM7/977-2 laminates.

the mechanical properties of composites. Therefore, intermediate modulus carbon fiber was employed for cryotank application [29, 30].

Toughened resins were developed primarily for aircraft applications to improve the compression-after-impact strength (CAI) of composite structures. They are also preferred for liquid hydrogen tanks because of greater impermeability after thermo mechanical cycling. For example, 8552 epoxy resin, which was made by Hercules, 977-2 and 5320-1resins, which were made in ICI, were used widely in LH2 cryotank fabrication.

Toughened resins appeared to be generally more resistant to ignition; therefore, toughened resins were used in LO2 tank fabrication. LOx compatibility is another issue that should be considered for Lox tank.

CF/977-2 prepreg was used widely in cryogenic applications because of excellent toughness and processing, for example, X-33 LH2 tank and 10 m composite demonstrator tank made in CCTD phase I [31, 32]. CYCOM 977-2 is a 177°C curing toughed epoxy resin. It is formulated for autoclave or press molding. Unidirectional tape and woven fabric impregnated with CYCOM 977-2 resin will retain tack for at least 10 days and has a long mechanical out life suitable for fabrication of large structures.

Table 3 showed the properties of IM7/ 5320-1, IM7/977-2, and IM7/8552 laminates. Tensile property, compressive property, and shear property of them were basically equivalent, the CAI of IM7/ 5320-1 were the lowest. The advantage of 5320-1 is that it requires no autoclave curing.

5. Conclusions

Composites are seen as one of the key components in the drive by NASA and the aerospace industry to decrease the weight of future launch vehicles as a means of reducing the cost of launching payloads.

NASA and Boeing have made significant progress in the US within the Composite Cryotank Technology Demonstration (CCTD) Project in which a large-scale composite liquid-hydrogen cryogenic tank has been designed, built, and tested under relevant flight loads.

For future space missions, there is still work to be done in producing out-of-autoclave tanks that meet the stringent permeability requirements.

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