

Permeability Test Measurement of Composite Material for Reusable Launching Vehicles (RLV)

Charanjit Singh, Karthik Sunil, Prudhvinath Reddy, Idris Syed Mahaboob, Raja Sekhar Dondapati

School of Mechanical Engineering, Lovely Professional University, Punjab-144411

charanjitsingh412@gmail.com, karthiksunil.09@gmail.com, prudhvi143mountaindew@gmail.com,

idrismec@gmail.com, rajasekhar9@gmail.com

Abstract: Polymer composites are replacing metals over the time due to their light weight, high strength to weight ratio, excellent physical and thermal properties. Hence, they are ideal candidates where weight is of particular concern. One such application is reusable launch vehicles (RLV). Use of polymer Composite materials instead of conventional metal alloys for fuel tank reduces weight of launching vehicle so that more payloads can be carried into space, thereby reducing cost per kilogram of payload. Polymer composites at room temperature show familiar properties but when they are exposed to low temperature, properties change rapidly and show abnormal behavior. Material becomes brittle as temperature is lowered and fails even at low stresses. It becomes further complex when fibrous polymeric composites are used at cryogenic temperatures. In this context, the suitability of polymer composites at low temperature in RLV has to be addressed since during fuelling with liquid hydrogen, the temperature of tanks drops to cryogenic temperature (20K). Polymer matrix composites are well-known class of engineering plastics, capable of withstanding such extreme thermal cycling and low velocity impacts, while retaining their impermeability.

Keywords— Composite material, Universal Testing Machine, Cryogenics, Load cell.

I. Introduction

Composites find numerous structural applications in the aerospace industries due to their low weight, high strength to weight ratio and good thermal and mechanical properties. In recent times composite materials find applications in the design of cryogenic fuel tanks for reusable launching vehicles (RLV). The structural systems of these vehicles are subjected to severe thermal and mechanical loading since they contain cryogenic liquids like LH₂ and LOX. Due to the thermal and mechanical loading, transverse cracks are induced and create a path for cryogenics to flow between the laminates of composites. As LH₂ is highly combustible, forms a combustible mixture when it comes in contact with Air/O₂ and results in hazards for RLV. The permeability of the composite should be kept minimal to avoid such hazards. The cryogenic tanks are investigated as an integrated tank system. An integrated tank system includes the tank wall, cryogenic insulation, Thermal Protection System (TPS) attachment sub-structure, and TPS. In this context it is very important to investigate the effects of permeability on the fuel tank made up of composites. Also, the effects due to mechanical load and pressure need to be

investigated. As a whole, the composite must withstand mechanical and thermal loading.

II. Literature Survey

Ray W. Grenoble and Thomas S. presented the experimental methods and results of the correlation between damage state and hydrogen gas permeability of laminated composite materials under mechanical strains and thermal loads [1]. A specimen made from composite material has been mechanically cycled at room temperature to induce micro crack damage. Leak rate was found to depend on applied mechanical strain, crack density, and test temperature. Hydrogen gas was supplied along the free edges of the specimen. Tensile modulus was found to be decreased, and micro crack damage increased, as the number of cycles increased. Crack density, as a function of ply orientation, was measured and found to correlate with the leak of hydrogen gas through the specimen. Tension modulus and strength were measured at room temperature, -196°C and -269°C on five different specimen ply lay-ups. Specimens were preconditioned with one set of coupons being isothermally aged for 576 hours at -184°C, in an unloaded state. Another set of corresponding coupons were mounted in constant strain fixtures such that a constant uniaxial strain was applied to the specimens for 576 hours at -184°C. A third set was mechanically cycled in tension at -184°C [2, 3]. The measured properties indicated that temperature, aging and loading mode can all have significant influence on performance. Moreover, this influence is a strong function of laminate stacking sequence. For tension loading, longitudinal, transverse stiffness and strength decreased as the test temperature decreased. Conversely, the tensile shear modulus and strength increased as the temperature decreased. Damage such as transverse matrix cracks (TMC) and delaminations are prone to develop in composites well below the load levels that would result in mechanical failure [4]. This microscopic damage leads to a leakage path for the fuel. The leakage is influenced by many factors, including pressure gradients, micro crack density, connectivity of the cracks, residual stresses from manufacture, service-induced stresses from thermal and mechanical loads and composite stacking sequence. It is expected that there is a direct relationship between leakage and damage opening but the connectivity of matrix cracks is also a major factor affecting the leakage. In this article, the leakage rate through the damage network is discussed based on earlier studies for the opening of damage

paths due to TMC and delamination, including the TMC intersection area.

Leak characteristics of CFRP (carbon fiber reinforced plastic) cross-ply laminates are experimentally investigated under biaxial loadings using a specially prepared in-plane biaxial testing system [5]. Permeability through the damaged laminate under biaxial stresses was measured with a leak detection system. Experimental results reveal that leakage through the damaged laminate is in correlation with the amount of damage and depends not only on load level but also on biaxial load ratio. Leak analysis, which had been previously developed, was employed and calculations of permeability were in good agreement with the experimental data. Prior to measurement of leaks, the cruciform specimens had been preloaded with cyclic biaxial tension to induce matrix cracks. The results of the leak analysis show that crack opening displacements, due to the thermal and mechanical loads, dominantly determine the leak level under biaxial loadings, and the damages in the laminates, which include the crack densities and in-plane defects at crack intersections, also affect the leakage properties. The behavior of a high temperature carbon / polyimide composite, in a cryogenic environment was studied [6]. The results were compared to results from similar testing of a carbon / epoxy and a carbon / bismaleimide composite in order to begin to scale the difficulties involved with using high temperature polymer matrix composites (PMCs) in cryogen containers on launch vehicles. The composite developed transverse cracks in all plies after fewer cycles than a carbon / bismaleimide when each was cycled between -196°C and their respective maximum service temperatures. A change to a cross-ply resulted in a substantial reduction in the tendency toward a complete crack network in past work. However, the same change from a $[0/45/-45/90]_S$ lay-up to a $[0/90]_{2S}$ lay-up produced only a small delay of 50 cycles in the formation of transverse cracks in all plies of the composite samples. On the other hand, the lay-up change corresponded to a large permeability reduction possibly due to less delamination in the $[0/90]_{2S}$ samples or due to some of the transverse cracks remaining near the sample edges. Previous cryogenic cycling research has focused on improving understanding of the mechanisms that lead to a leakage-producing network of cracks in carbon / epoxy and carbon / bismaleimide composites and to evaluate a number of materials for use in cryogenic pressure vessels [7]. However, the large fuel tanks and other cryogenic components of future reusable launch vehicles may benefit from the use of even higher temperature composite materials through the reduction in the weight of the thermal protection system needed to protect the composite components inside the vehicle. Trends indicate that implementing high temperature PMCs in cryogenic components of reusable launch vehicles to reduce TPS requirements will most likely require more careful material, lay-up, and service temperature profile considerations than those for implementing medium temperature PMCs. Experiments were performed to investigate the effect of cryogenic cycling on the gas permeability of various composite laminates for cryogenic storage systems [8]. It was found that the textile composites have lower permeability than laminated composites even with increasing number of cryogenic cycles.

Nano-particles dispersed in one of the ply-interfaces in tape laminates do not show improvement in permeability. Micrographs of sections of various specimens provide some insight into formation of microcracks, and damage before and after cryogenic cycling. In laminated tape composites microcracks in various layers connect and form an easy path for gas leakage. Composites wherein plies of different orientations are dispersed rather than grouped show excellent performance even after cryogenic cycling. In textile composites the damage is restricted to regions contained by the weave yarns and hence the permeability does not increase significantly with cryo-cycling. The permeability test facility was constructed following the standard test method documented in ASTM D14382 (Re-approved 1997). The permeability of laminated composites was found to increase between 3% to 12% after cryogenic cycling. The rate of hydrogen leakage can be a function of the material used, method of fabrication used to manufacture the tank, mechanical load, internal damage-state of the material and the temperatures at which the tank must operate [9]. LHe and LH2 test results indicate that further research is required to determine the acceptability of LHe leak tests as screening tests when LH2 leaks are the primary concern. Test results also indicate that measured LH2 leak rates are above acceptable levels for a SSTO LH2 tank and that they can vary widely with load level and location in the structure. This investigation has also demonstrated the need for structures-level leak testing to validate composites and composite manufacturing processes for LH2 tank applications. A cryogenic thermal -cycling device, or cryocycler, was designed and built to efficiently provide extreme temperatures at either end of the spectrum to the specimens [10]. Groups of narrow coupon laminates were exposed to various numbers of cycles and then test to reveal any degradation of the mechanical properties. The paper explored the mechanical properties of the composite material after various levels of cycling at extreme temperatures. The specimen did not exhibit any crack growth or apparent loss in stiffness or strength properties after 10 cycles of the simulated flight cycles. The only observable change was in failure mode, which did not seem to affect any of the other properties measured. Further testing of this material is recommended at higher numbers of cycles to find where the material begin to crack, and resolve the ambiguity in the critical strain energy release rate..

III. Test Methods for Permeability

The permeability is defined by the amount of gas that passes through a given material of unit area and unit thickness under unit pressure gradient in unit time. The SI unit of the permeability is $\text{mol/s/m}^2/\text{Pa}$. The standard test method for determining gas permeability is presented in ASTM D14382 (Re-approved in 1997) "Standard Test Method for Determining Gas Permeability Characteristic of Plastic Film and Sheeting". The permeability can be measured by two experimental methods, manometric determination method and volumetric determination methods.

Manometric determination method

The experimental setup for the manometric determination method is shown in Fig. 1. The lower pressure chamber beneath the specimen in Fig. 1 is initially vacuumed and the transmission of the gas through the test specimen is indicated by an increase in pressure. The manometric method of permeability determination is tedious since mercury compounds are used while experiment requires special handling procedures and safety. Generally this method is avoided for determination of permeability.

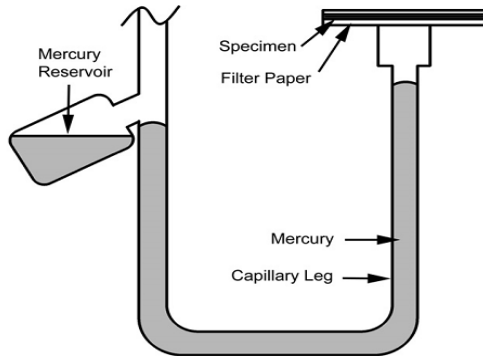


Fig 4. Permeability determination for manometric method.

Volumetric determination method

The permeability can also be measured using volumetric determination as shown in Fig. 5. The lower pressure chamber is exposed to atmospheric pressure and the transmission of the gas through the test specimen is indicated by a change in volume. The gas volume flow rate is measured by recording the rise of liquid indicator in a capillary tube per unit time. The permeability determination by volumetric method is more sophisticated and easy to handle. Nettles [9, 13] investigated the effects on the volume-flow rates by using various types of liquid. The volume-flow rates obtained using water, alcohol and alcohol with PhotoFlo were not significantly different. Methyl alcohol is generally chosen as the liquid indicator since alcohol has low viscosity and density. The methyl alcohol is normally colored with a dye to obtain precise readings on the scale marks.

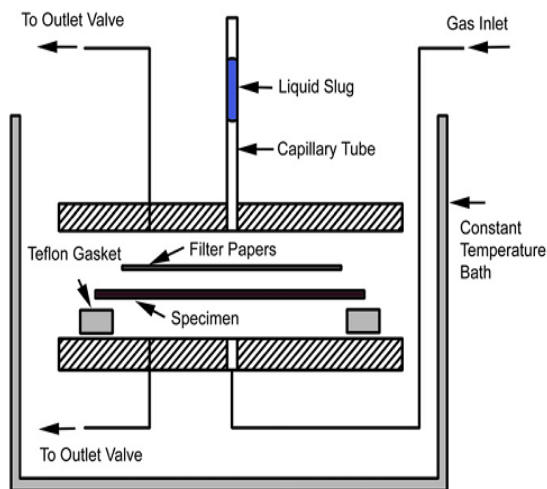


Fig. 5. Permeability determination for volumetric method.

The gas transmission rate (GTR) is calculated using the ideal gas law. The permeance is determined as the gas transmission rate per pressure differential across the specimen. And, then the permeability (\bar{P}) is determined by multiplying permeance by the specimen thickness.

$$\bar{P} = \text{Permeance} \times \text{Specimen Thickness}$$

In order to have error free reading, ambient temperature and pressure are to be noted while doing experiment and average values are to be taken.

The permeability experimental apparatus basically consist of two chambers between which specimen has to be placed as shown in fig. 5. The permeant gas is pressurized in the upstream chamber. The gas permeates through one side of the specimen and escapes out of the other side. The escaped gas is collected in the downstream chamber and enters into a glass capillary tube. The amount of gas escaping per unit time is measured. The permeance is determined by gas transmission rate and the pressure differential across the specimen. The gauge pressure of the gas in the upper upstream chamber is measured using a pressure transducer. The ambient Pressure is measured by a barometric sensor. A precision pressure regulator provides constant gas pressure to the upstream chamber. The ambient temperature is measured using a glass capillary thermometer. The specimens are mounted horizontally between the upstream and downstream chambers and clamped firmly by applying a compressive load. The specimen is sealed with a gasket and an O-Ring. A force gauge mounted at the top measures the compressive load to ensure that the same amount of compressive load is applied on the specimens for every test. The compressive load should be enough to prevent gas leakage, but should not damage the specimen. The upstream chamber has an inlet vent and an outlet vent. The inlet vent allows the gas flow into the upstream chamber and the outlet vent is used to purge the test gas to atmosphere. The down- stream chamber has two outlet vents. One is used to purge the test gas to atmosphere and the other allows the gas flow to the glass capillary tube for measurements. The sensitivity of permeability measurement can be improved by increasing the gas transmitting area of a specimen.

The glass capillary tube is mounted horizontally to minimize the gravity effect on the capillary indicator and for easy reading of the scale marks on the capillary tube. Nettles [9] found that there was no significant difference in the volumetric flow rate when the capillary tube is placed vertically or slanted. A magnifying glass is used to read the scale marks at the top of the meniscus of the liquid indicator. The liquid indicator in the glass capillary tube is used to measure the rate of rise of the liquid indicator. The rate is used to calculate the volume-flow rate of the escaped gas across the specimen. The primary objective of this investigation is to study the hydrogen permeability of laminated composites. However, hydrogen gas is highly flammable and explosive when it mixes with air, and it needed to be handled with extreme care during the test. Hence, other permeate gases were considered as a substitute

for the hydrogen gas. The molecular diameter of various gases is listed in Table 1. To choose a permeate gas, the molecular diameter determined from viscosity measurement is mainly considered since the permeability is related with the volumetric flow rate directly. Since helium has the smallest molecular diameter, its apparent permeability is higher than that for other gases, and it will yield a conservative value for permeability.

Table 1 Molecular diameter of various gases from CRC Handbook of Chemistry and Physics, 54th Edition[12]

Type of Gas	From Viscosity	From Molecular Diameter (cm)	
		From Vander Wall's Equation	From Heat Conductivity
Helium	1.9×10^{-8}	2.6×10^{-8}	2.3×10^{-8}
Hydrogen	2.4×10^{-8}	2.3×10^{-8}	2.3×10^{-8}
Nitrogen	3.1×10^{-8}	3.1×10^{-8}	3.5×10^{-8}

IV. Conclusions

- Permeability is found to be dependent on tensile load, cracks, thermal cycling and strength of composite.
- For multiple leaks in a composite, the leak rate can be calculated using overall conductance. Conductance in series and parallel are calculated and then summed to get total conductance
- The permeability test under bi-axial loading shows increase in permeability with tensile load.
- The permeability is increasing with each thermal cycling but becomes constant after a particular no of thermal cycles.
- Fracture load decreases due to material degradation at cryogenic temperatures.
- Fracture toughness is increasing with the increase in middle layer thickness.
- Stacking sequence also plays a vital role in determining properties of composites
- Presence of inclusion results in sudden failure of material.
- Presence of crack or initiation of crack results in leakage and proportionate propagation of the same.

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