Polymer Lined COPVS Formed using an Integrally Heated Rotational Moulding Tool and Laser Assisted Tape Placement

POLYMER LINED COPVS FORMED USING AN INTEGRALLY HEATED ROTATIONAL MOULDING TOOL AND LASER ASSISTED TAPE PLACEMENT

Brendan R. Murray[1, 2], Sean B. Leen[1], Christopher O.A. Semprimoschnig[3], Conchúr M. Ó Brádaigh[4]
[1]Mechanical Engineering, National University of Ireland Galway, IRELAND
[2]MaREI Centre, Environmental Research Institute, University College Cork, IRELAND
[3]ESA-ESTEC, Materials Space Evaluation and Radiation Effects, THE NETHERLANDS
[4]Institute for Materials and Processes, University of Edinburgh, SCOTLAND, UK

ABSTRACT

Composite overwrapped pressure vessels (COPVs) are a critical component in space applications as their ability to store highly permeating fuels at high pressures under cryogenic conditions makes them an integral part of propulsion systems aboard rockets, satellites and spacecraft. Recent research has focused on replacing the standard metallic liner, in a COPV, with a polymer liner to reduce costs. An integrally heated rotational moulding tool has been constructed and used to produce demonstrator polymer liner components. The integrally heated tooling provides improved control of heating parameters with segregated heating throughout the tool which increases the dimensional accuracy of the part while also reducing energy consumption. The permeability of prospective liner materials has been evaluated for use in a demonstrator COPV with results showing acceptable levels of fuel containment. Polymer liner samples have been overwrapped in a laser assisted tape placement (LATP) process with a CF-PEEK tape to form final COPV wall configurations. Cryogenic cycling of liner-overwrap samples has shown crack resistance over multiple cycles preserving the polymer liners barrier properties. These new and novel manufacturing techniques show promise for future COPV production using polymer liners and out-of-autoclave processes.

1. INTRODUCTION

1.1 Composite Overwrapped Pressure Vessels (COPVs)

COPVs have become a critical component in space applications since their initial introduction in the early 1970s [1, 2]. Their ability to store highly permeating fuels at cryogenic conditions has solidified their usage aboard satellites and spacecraft in propulsion systems, environmental control systems and life support systems [3-5]. Their significant reduction in weight, dimensional flexibility and inherent cost savings have established them as a viable alternative to all-metal pressure vessels with COPVs being regularly incorporated into current mission designs.

COPVs consist of two distinct layers, an inner low permeability liner and an outer high strength fibre overwrap. The thin inner liner contains the fuel and prevents leakage to the surrounding environment while the outer overwrap absorbs the stresses generated by the containment of the fuel within. Titanium has been the preferred liner material in aerospace applications due to its high specific strength, resistance to chemical attack and excellent barrier properties [6, 7]. Kevlar and carbon fibre composites have been the overwrapping materials of choice due to their high strength and low weight characteristics.

While titanium has been the preferred liner material, its main drawback is the associated cost. Precision machining of titanium liners can cost in the region of €80,000 ($90,000) for a spherical liner with a 500 mm (20 inch) diameter [8]. This high forming cost has been justified by the weight reduction achieved through the use of COPVs as the cost of sending payloads to space is estimated at €10,000 kg^{-1} to €17,000 kg^{-1} ($5000 lb^{-1} to $8000 lb^{-1}) [9, 10]. However these high costs have hindered the incorporation of COPVs in other industries and highlights the need for alternative low cost materials.

1.2 Background

Recent research has focused on polymer materials as a viable replacement for metal liners in COPV applications. Polymers are low-cost, low weight, and easy to handle materials, with low permeability properties and a high resistance to chemical attack, ensuring good compatibility with highly permeating fuels. A number of projects have already been conducted with varied success in regards to incorporating polymer liners in COPV designs. Following the failure of the X-33 composite cryogenic tank, initial research focused on the testing of polymer liner materials as a low permeability barrier for cryogenic fuel containment [11]. AVIO, an Italian based manufacturing company, conducted extensive research into prospective liner materials. A thermally-welded polymer liner was produced and overwrapped with a tape-placed composite and subsequently tested as a liquid oxygen storage unit [12]. AIRBUS Defence & Space has also conducted significant work in the area, producing a spherical 300 Litre polymer-lined tank with a cost saving of over 30% compared to other models [13]. They also continued this work with an aim to incorporate polymer liners in larger upper stage tank structures with the testing of a number of polymer materials conducted [14].

1.3 Overview

The major issue with these recent advancements in polymer lined COPVs is the capital intensive nature of the manufacturing methods chosen (injection blow moulding, thermal welding, etc.). The need for an alternative low cost manufacturing method is crucial for future polymer-lined COPVs if they are to be cost effective. To this end, a modified rotational moulding process is presented in this paper as an alternative manufacturing method to produce demonstrator polymer liners. Helium permeability testing of polymer liner materials formed from base powder materials has been undertaken to determine the ability of these materials to store highly permeating fuels. These permeability tests are followed by the overwrapping of polymer liner samples with a laser assisted tape placement process. These samples have been cryogenically cycled in liquid nitrogen with X-Ray CT scans taken to assess the ability of the liner-overwrap configuration to resist crack growth in the wall cross section.

2. EXPERIMENTATION

2.1 Integrally Heated Rotational Mould Tooling

The modified rotational moulding process, displayed in Fig. 1, utilises the original principles of rotational moulding [15] but replaces the heating oven with an integrally-heated mould tool contained within the rotating axes [16]. The mould tooling is electrically powered via slip ring connections in the joints, which transfer power through the rotating arms. The mould itself is constructed with heating coils uniformly distributed around the mould to allow for direct heating and improved thermal control [17]. The modified tooling allows for multiple temperature readings at critical locations around the mould during processing, while the placement of the heating coils around the tool allows for increased control of temperature distributions. This gives a significant improvement in the dimensional accuracy of the part with temperature distributions and energy consumption being reduced. This reduces the cost of running this process by localising the heating around the mould tooling and removing the energy losses associated with using a large oven in the traditional rotomoulding process [18].

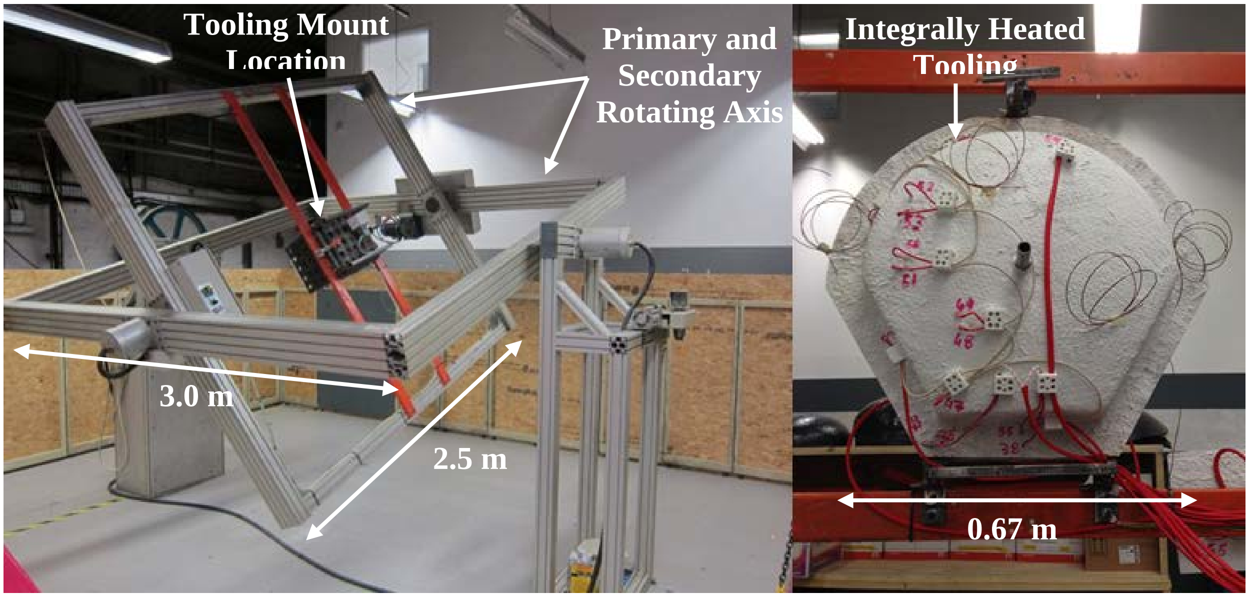


Figure 1. The biaxially rotating arms of the rotational moulding rig with the integrally heated tooling also pictured.

2.2 Helium Permeability Testing

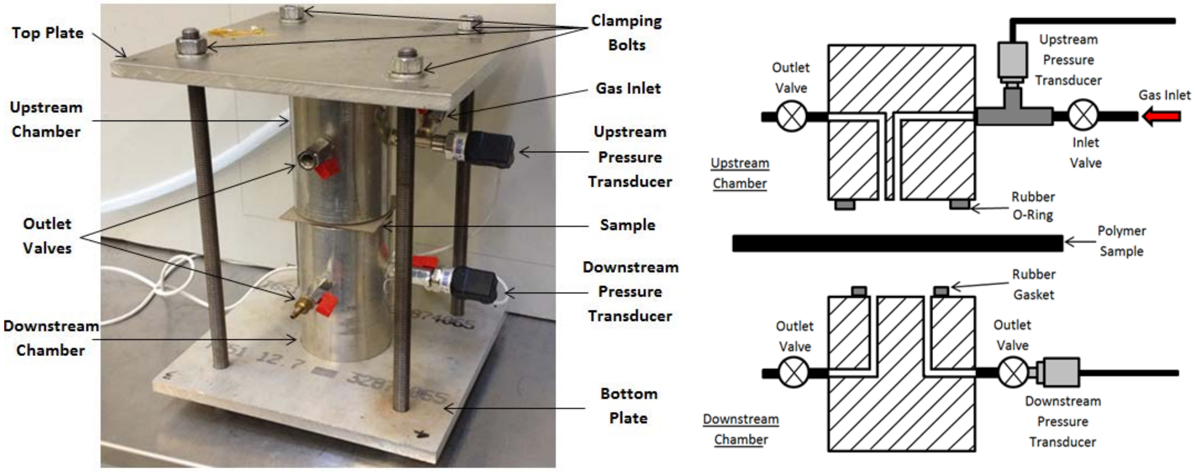


Figure 2. The permeabiltiy test rig used along with a schematic of the internal structure.

The permeability test apparatus used in this study is designed using a modification of the ASTM D1434 test standard [19]. This apparatus follows the same principles as the standard but instead of using a liquid slug indicator, a second pressure transducer has been placed in the downstream chamber to gauge the pressure increase over time. The permeability test rig, in Fig. 2, consists of two chambers, one upstream and one downstream of the flow, with a sample placed between the chambers and clamped with a constant force. Helium gas is supplied to the upstream chamber at a constant pressure and the pressure increase in the downstream chamber is monitored over time. The permeating gas diffuses through the sample and the increase in gas pressure in the downstream chamber is used to calculate (i) the leak rate through the sample in units of scc/m^{2}s and, hence (ii) the permeability coefficient (scc/m.s.bar). Three samples from seven materials have been tested here for helium permeability; four grades of PEEK powder (Victrex 150P and 150PF, and Evonik 1000P and 2000P) along with single grades of PA11, PA12 and PVDF supplied by Matrix polymers. A pressure difference of 2 bar has been employed for all tests with sample thicknesses ranging from 3 to 5 mm.

2.3 Laser Assisted Tape Placement (LATP)

Laser assisted automated tape placement is an out-of-autoclave processing method for the production of thermoplastic composite parts using a robotic arm, a laser welding head and a wheel fed strip of composite tape [20]. It uses a thin 13 to 14 mm wide carbon fibre thermoplastic tape that is built up in multiple passes and layers to create a finished part of the desired shape, size and orientation. The LATP unit consisted of a robotic arm (KUKA KR 180 R2900) with six axes of motion. The tape heating is assisted through the use of a laser-line diode laser module (LDM) 3000 W system (supplied by Advanced Fibre Placement Technology (AFTP) GmbH).

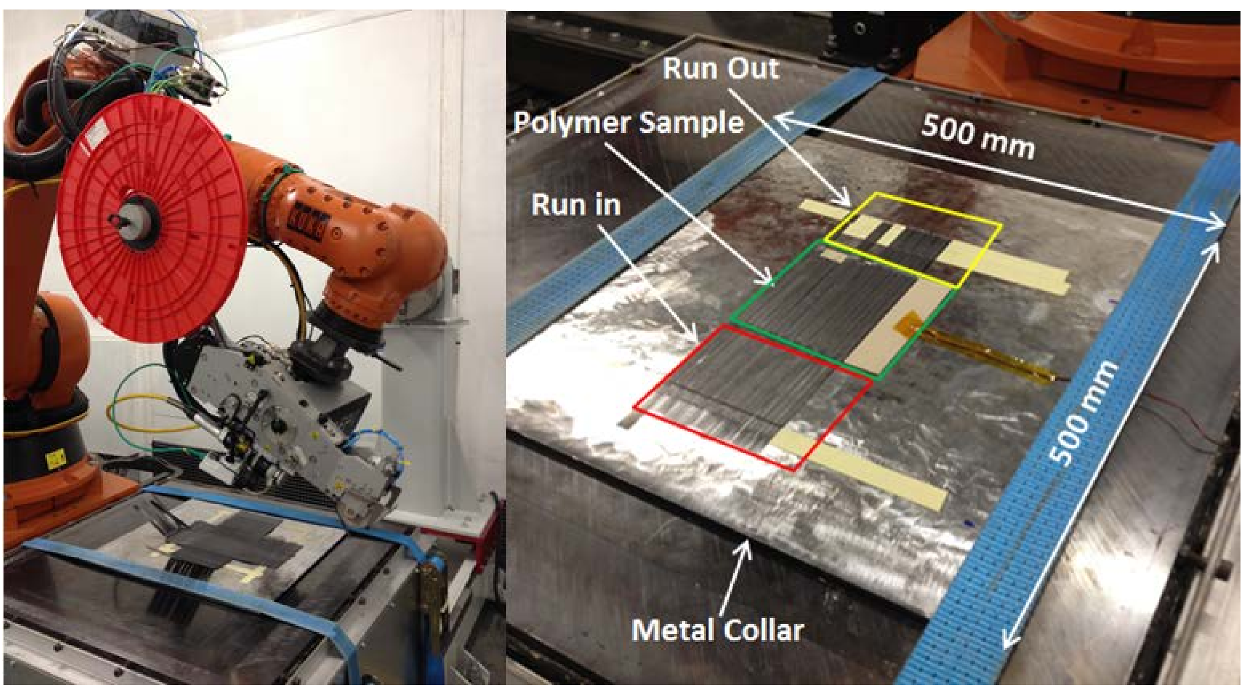


Figure 3. The LATP unit used and the overwrapping methodology for carbon fibre thermoplastic tape laying over polymer liner samples.

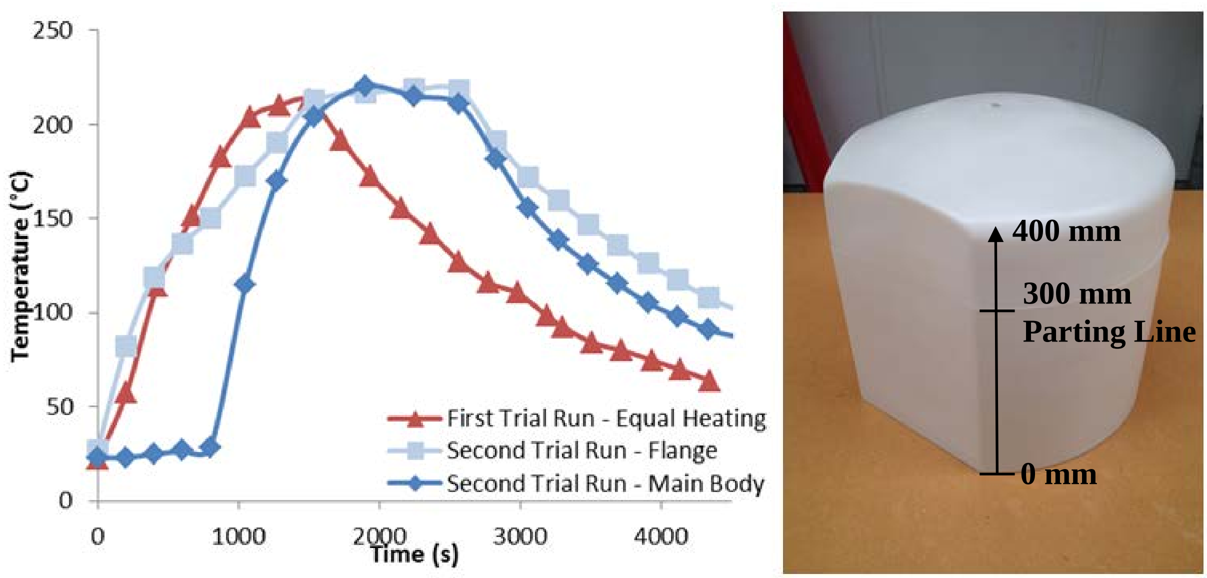
A mild steel plate, Fig. 3, was used to accommodate the run in and run off of the LATP process as it takes a nominal length of 150 mm of tape for the processing conditions to reach equilibrium conditions before the process becomes uniform. The steel plate holds the polymer sheet (nominal dimensions of 140 mm *x* 140 mm *x* 5 mm) and allows for uniform and concise tape placement over the polymer sample. It also prevents the introduction of unnecessary height variations between the samples and the laser welder and consolidation roller, which mitigates the subsequent formation of defects. A 5 mm thick PEEK 150PF sample has been overwrapped with eight layers of CF/PEEK tape in a $[0/90/0/90]_S$ pattern as a demonstration of the process' capabilities. Samples were then cut from this panel for cryogenic cycling and X-Ray CT analysis.

2.4 X-Ray CT Scanning

X-Ray CT scanning has been used to analyse the internal structure of LATP joints which have been cycled to cryogenic temperatures. It uses an X-ray gun to take over one thousand individual images of a sample and then arranges and regenerates the collection of single images into a solid three dimensional structure with internal voids and defects highlighted. The X-Ray CT machine used here, and housed in the ESA ESTEC facility in the Netherlands, was a GE V/tome/X m300 with a Microtom gun operating at a voltage of up to 230 kV giving a high resolution of as little as 1 μm in size (depending on material properties, setting parameters and sample size). The rendering software used here is VG Studio Max 2.2 and this has been used in conjunction with the "defect analysis" tool to perform internal analyses of cycled LATP joints. These internal analyses were used to assess the extent of crack growth in PEEK liners which had been overwrapped with CF/PEEK tape and cryogenically cycled in liquid nitrogen.

3. RESULTS

3.1 Modified Rotational Mould Tooling Controls



(a) **(b)**

Figure 4. (a) Heating cycles for the part formation to highlight the improvements in flange control achievable and (b) an image of a formed polymer demonstrator.

The effects of segregated heating zones have been highlighted through the manufacture of demonstrator parts showcasing the effects of temperature control on part consistency. Two different temperature cycles, shown in Fig. 4, were used to highlight the difference in flange heating control optimization to improve the quality of the wall thickness distribution around the parting line. The first trial run for the integrally-heated tooling shows the effects of heating the flange and main body at the same rate for the entire cycle. The second trial run utilises an optimised flange heating approach. The designation of the flange as a single zone allows for heating of the flange to occur at a faster rate, or earlier in the heating cycle, promoting wall thickness build up around the parting line (the area of greatest heat loss) and keeping the temperature distribution uniform across the wall section of the tool during the forming process.

The wall thickness measurement results shown in Fig. 5 show that at the parting line (300 mm mark on the distance scale) the optimised flange heating has created a more uniform wall distribution across the entire wall length. This is beneficial for part thickness consistency while also showcasing the tooling ability to control wall thickness distribution.

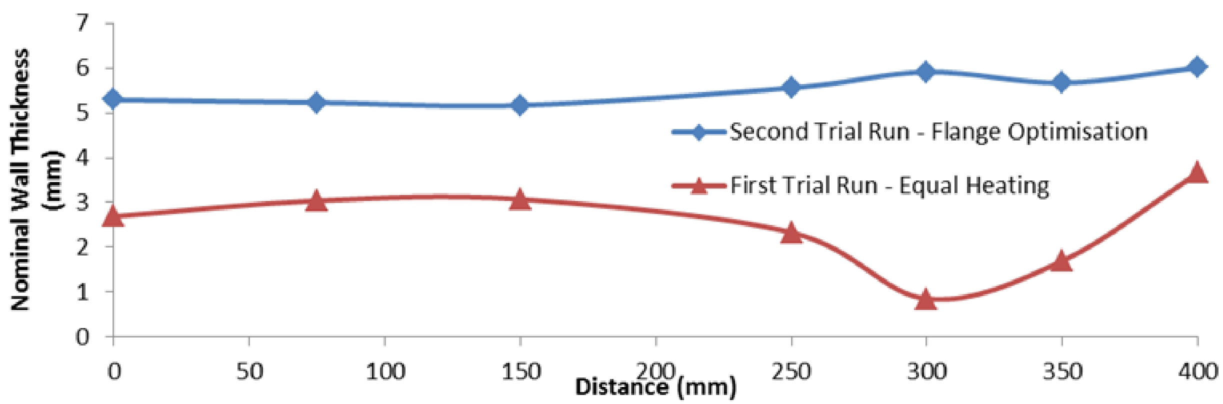


Figure 5. Wall thickness distributions along the demonstrator length for two different heating cycles with significant differences visible at the parting line (300 mm mark).

3.2 Permeability Results

The leak rates for three samples of each material were tested, with the permeability coefficients for each material presented in Fig. 6. From these results it is clear that, of the materials tested, PA11 and PVDF significantly outperform PEEK and PA12, with low permeability coefficients of approximately 6×10^{-7} scc/m.s.bar. PA12 and PEEK materials have permeability coefficients of 1×10^{-6} scc/m.s.bar or greater with values reaching approximately 1.4×10^{-6} scc/m.s.bar for the PEEK 2000P material.

These results can be used to predict the leak rates of future COPVs whose operating conditions are known. For the current project the envisaged usage is for a 90 L cylindrical tank with domed ends (corresponding to a 1 m^2 internal surface area), an operating pressure of 5 bar, and a maximum allowable leak rate of 1×10^{-3} scc/s. Fig. 7 shows the predicted leak rate for different liner thicknesses for the as-tested materials, showing that all materials are able to achieve the low leak rate requirements at liner thicknesses exceeding 5 mm. This is the first indication that polymer liner materials tested here have the required functionality to meet the low permeability requirements of COPV applications.

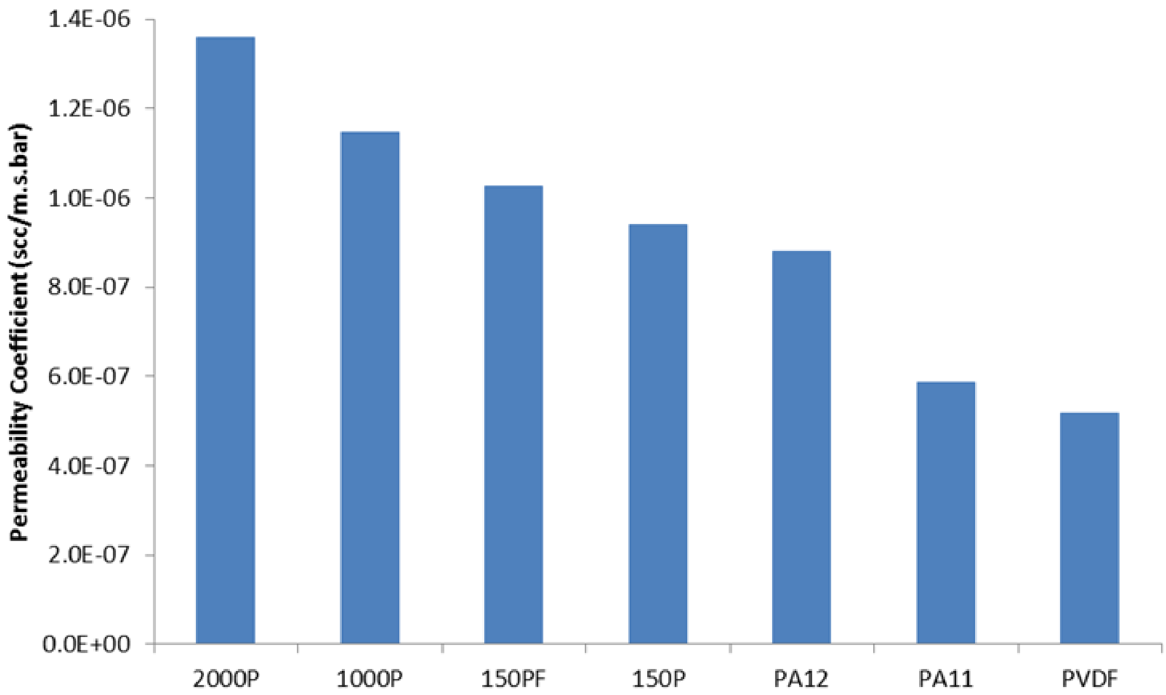


Figure 6. Permeability coefficient results for all polymer materials tested.

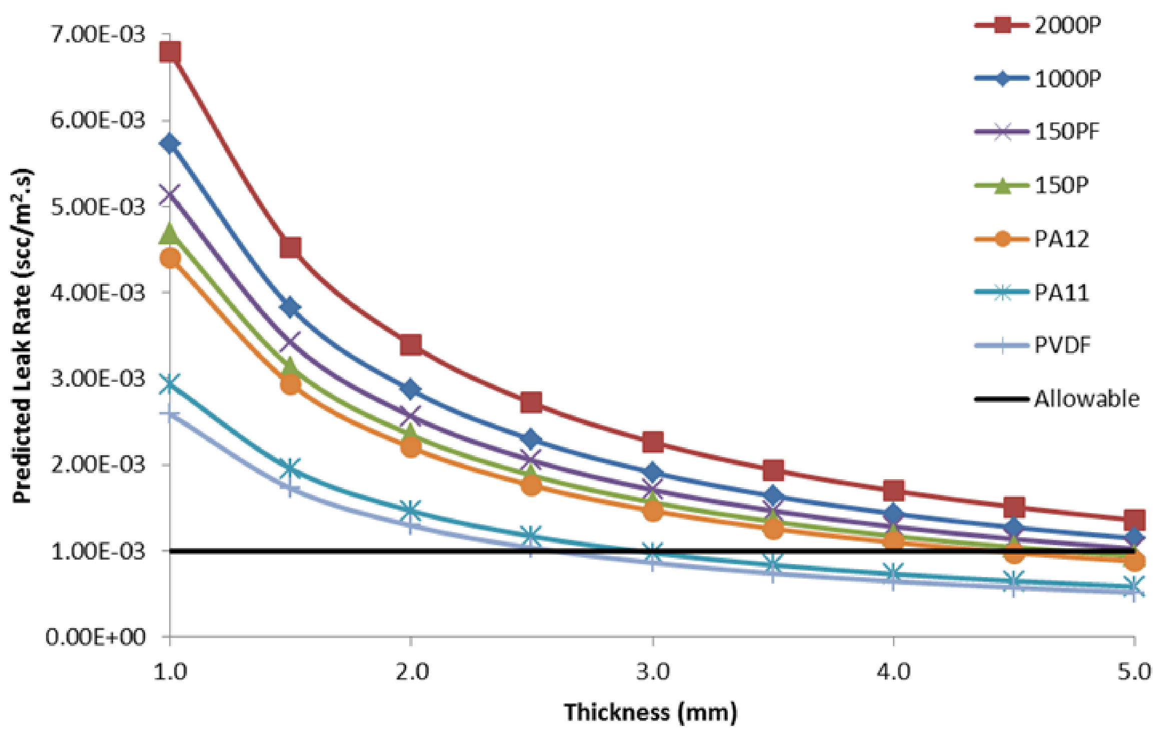


Fig. 7. Predicted leak rates for a 1 m^2 area of each material with a 5 bar pressure difference for varying liner thicknesses.

3.3 Cryogenic Cycling of LATP Samples

The third part of the testing conducted involves an assessment of the overwrapping process on the polymer liner and cryogenic environmental testing of the final polymer lined COPV layup. As has already been outlined, a PEEK 150PF polymer sample has been overwrapped with a CF/PEEK tape using the defined LATP process. This is indicative of the final manufacturing methods to be used for forming the COPV designed here and so is a good representation of the expected results for the future COPV designs. A cross sectional image of the CF/PEEK tape on a PEEK substrate has been included in Fig. 8 with the different liner-overwrap regions highlighted. There is no evidence of a significant weld zone from the LATP process with this microscopic techniques.

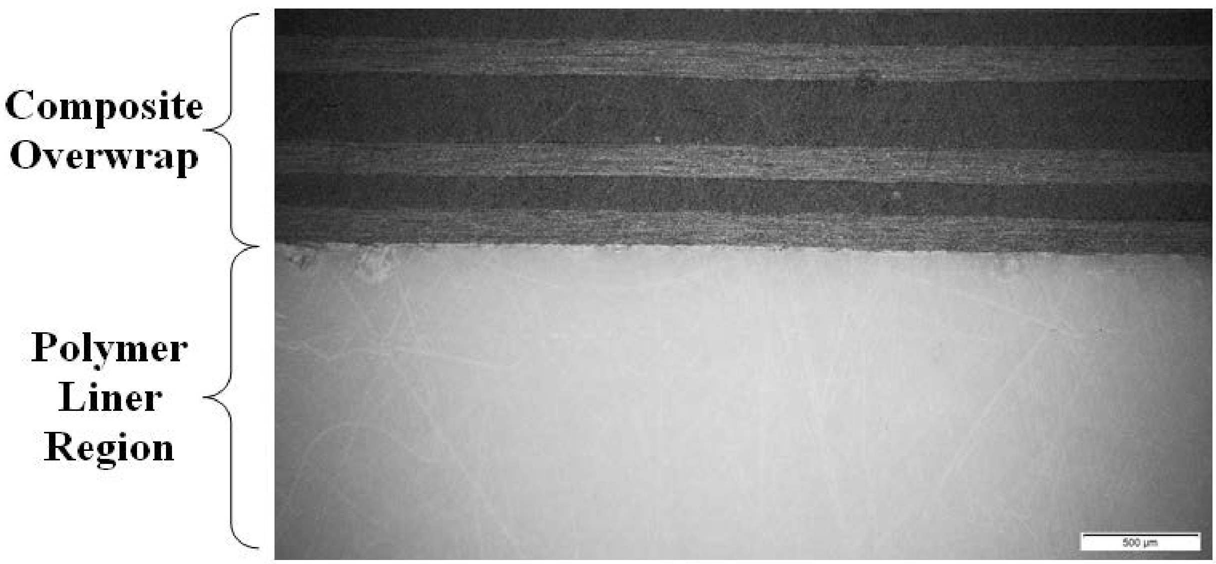


Figure 8. Microscopy image of the liner-overwrap cross section in a PEEK 150PF liner overwrapped with 8 layers of CF/PEEK tape.

The consolidation of the CF/PEEK tape on the polymer liner was successful, with visual inspection showing good bonding of the tape to the liner surface. X-Ray CT scans of the individual bond regions did show small regions of debonding between the CF/PEEK tape and the PEEK polymer surface as evidenced in Fig. 9. While this is a concern in regards to achieving a strong consistent bond across the two individual layers, it has not significantly hindered the subsequent cryogenic cycling trials as shown in Fig. 10. Here a PEEK 150PF sample overwrapped with CF/PEEK tape has been cycled in liquid nitrogen and intermittently scanned by X-Ray CT prior to cycling and after 1, 2, 3, 5, and 10 cycles. The samples retained the bond strength between the liner and overwrap over ten cycles, documented with no further debonding of the overwrap from the liner surface. In relation to crack growth, results were also positive for up to 10 cryogenic cycles, with no cracks observed in the majority of samples meaning that the liner configuration would retain its low permeability properties for up to 10 cryogenic cycles.

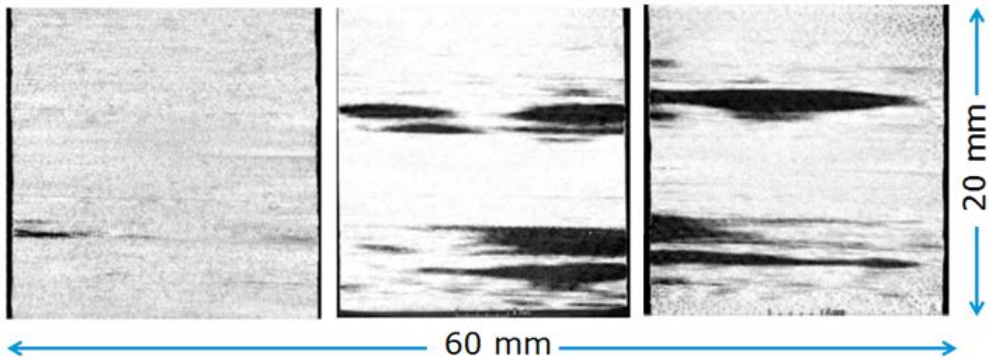


Figure 9. X-Ray CT images of debonding regions between the CF/PEEK tape and the PEEK 150PF liner surface highlighted by the darker regions signifying air gaps in the bond region.

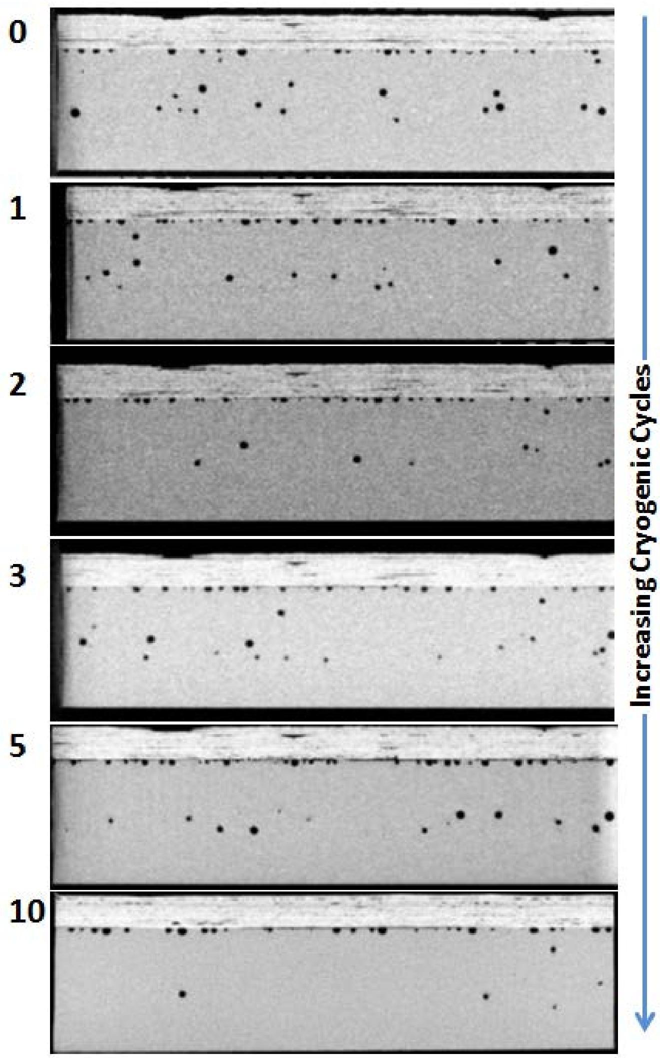


Figure 10. Cross sectional X-Ray CT images of a cryogenically cycled $[0/90/0/90]_S$ CF/PEEK to PEEK 150PF LATP laminate before cycling and after 1, 2, 3, 5 and 10 cycles with no micro-cracks present.

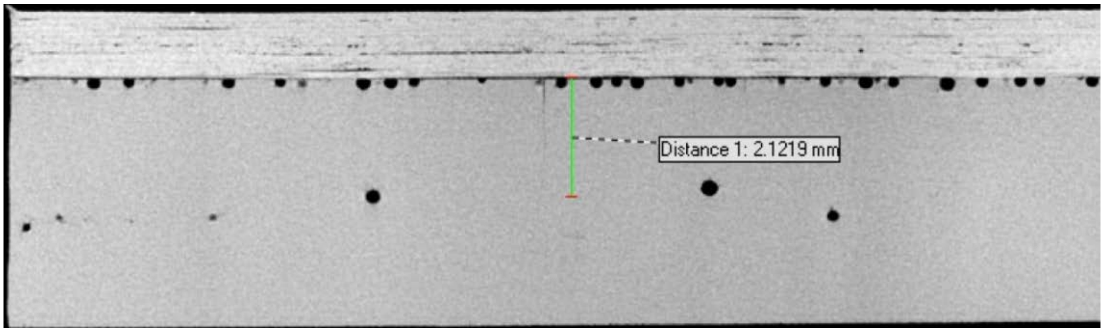


Figure 11. A 2 mm deep microcrack through the thickness of the polymer liner region in a $[0/90/0/90]_S$ CF/PEEK to PEEK 150PF LATP laminate after 50 cycles.

However after a further 40 cycles (giving a cumulative 50 cryogenic cycles) the samples did experience microcrack growth in the polymer liner region as evidenced in Fig. 11. Although this is an issue with respect to extended cycling applications, these results are a positive outcome for the use of polymer lined COPVs for up to 10 cycles.

4. CONCLUSIONS

A modified rotational moulding process has been designed and presented for the moulding of polymer liners for future COPV applications. It offers improved control in regards to wall thickness distributions via segregated heating lines, which has been proven through demonstrator part production and analysis of wall section thickness consistencies. A number of polymer materials have been tested with a helium permeability test rig and have shown low leak rates and low permeability coefficients. Specifically, PA11 and PVDF have outperformed all other materials tested with respect to low permeability coefficient measurements. Predictions based on these measured values have shown that, for the envisaged COPV designed here, polymer liners with a minimum wall thickness of 5 mm will provide sufficiently low rates of permeation at the maximum expected operating pressure, hence the materials can be used as acceptable replacements for liner materials in COPV applications. Laser-assisted tape placement has shown the capabilities needed to overwrap polymer liner samples in an out-of-autoclave process that provides a sufficient level of bonding between the liner sample and overwrapping tape. Cryogenic cycling of the liner-overwrap joints formed here with the LATP process have shown resistance to microcracking in the polymer liner section for up to 10 cryogenic cycles, even if failure occurs after a further 40 cycles. These results have shown that a modified rotational moulding process can be used to produce polymer liners which can then be overwrapped with a laser-assisted tape placement process. The polymer liners tested here are predicted to provide a sufficiently low permeability and are capable of withstanding the cryogenic environment inherently created by the storage of these highly permeating fuels for a limited number of cryogenic cycles.

5. ACKNOWLEGEMENTS

The authors would like to thank the Irish Research Council (IRC) and the European Space Agency (ESA) for joint funding of this research under the Network Partnering Initiative (NPI) and for the use of facilities in ESA's ESTEC centre in Noordwijk. They would also like to thank Terry McGrail and David Jones at the University of Limerick (UL) for the use of the LATP unit.

Research collaborators include ÉireComposites Teo, the Irish Centre for Composites Research (ICOMP), and Airbus Defence & Space. They would also like to acknowledge the specific help and technical support provided by P.J. Feerick and Michael Flanagan of ÉireComposites Teo.

6. REFERENCES

1. Mizutani, Y., Sugimoto, S., Matsuzaki, R., and Todoroki, A. "Fundamental Study of Integrity Evaluation Method for COPVs by Means of Acoustic Emission Testing." *Journal of Acoustic Emission* 27 (2009): 89-97.
2. McLaughlan, P.B., and Grimes-Ledesma, L.R. "Composite Overwrapped Pressure Vessels, A Primer." *NASA/SP–2011–57*. Johnson Space Center, Houston, Texas, USA, 2011
3. Grimes-Ledesma, L.R., Phoenix, S.L., Beeson, H., Yoder, T., and Greene, N. "Testing of carbon fibre composite overwrapped pressure vessel stress-rupture lifetime." *ASC/ASTM 21st Annual Technical Conference of the American Society for Composites*, Michigan, USA, September 17-20, 2006.
4. Thesken, J.C., Murthy, P.L.N., Phoenix, S.L., Greene, N., Palko, J.L., Eldrigde, J., Sutter, J., Saulsberry, and R., Beeson, H. "A theoretical investigation of composite overwrapped pressure vessel mechanics applied to NASA full scale tests." *NASA/TM-2009-215684*. Ohio Aerospace Institute, Brook Park, Ohio, USA, 2009.
5. Grimes-Ledesma, L.R., Murthy, P.L.N., Phoenix, S.L., and Glaser, R. "A comparison of various stress rupture life models for orbiter composite pressure vessels and confidence intervals." *9th Joint FAA/DoD/NASA Aging Aircraft Conference*, Atlanta, USA, March 6-9, 2006.
6. Tam, W.H., Griffin, P.S., and Jackson, A.C. "Design and manufacture of a composite overwrapped pressurant tank assembly." *38th AIAA/ASME/SAE/ASEE Joint Propulsion Conference*, Indiana, USA, July 7-10, 2002.
7. Thesken, J.C., Murthy, P.L.N., and Phoenix, S.L. "Composite Overwrap Pressure Vessels: Mechanics and Stress Rupture Lifing Philosophy." *NASA/TM-2009-215683*. Ohio Aerospace Institute, Brook Park, Ohio, USA, 2009.
8. Tam, W., Ballinger, I., and Jaekle, D.E. "Review and History of ATK Space Systems Commerce - The Past 15 Years." *Internal Report. ATK Space Systems Commerce Division*, California, USA, 2008.
9. Angelo, J.A. *Space Technology*. Greenwood Press, Westport, Connecticut, USA, 2003.
10. Labriet, M., and Poluet, L. "The missing step to building a lunar spaceport." *AIAA SPACE 2013 Conference and Exposition*. California, USA, September 10-12, 2013.
11. Grimsley, B.W., Cano, R.J., Johnston, N.J., Loos, A.C., and McMahon, W.M. "Hybrid composites for LH2 fuel tank structure." *33nd International SAMPE Technical Conference*, Vol. 33. Seattle, Washington, USA, November 5-8, 2001.
12. Mataloni A. "Elastomeric liner for cryogenic application." *Internal Summary Report, AVIO - Comprensorio BPD*, NTEESA10007 Iss.:2, Italy, 2005.
13. Benedic, F., Leard, J.P., and Lefloch, C. "Helium high pressure tanks at EADS space transportation new technology with thermoplastic liner." *1st Symposium on Potentially*

Disruptive Technologies and their Impact in Space Programs, Marseille, France, July 4-6, 2005.

14. Claudel, S., Repellin, A., Jaguenaud, L., Lacour, D., Bergerot, A., and Defoort, B. "Advanced and affordable thermoplastic based composite tank for cryogenic fluid storage." *SAMPE Conference*, Washington State Convention Center, Seattle, USA, May 17-20, 2010.
15. Crawford, R.J., and Kearns, M.P. *Practical Guide to Rotational Moulding*. Rapra Technology Ltd. Queen's University, Belfast, Northern Ireland, UK
16. Murray, B.R., Leen, S.B., and Ó Brádaigh, C.M. Void distributions and permeability prediction for rotationally moulded polymers. *Journal of Materials: Design and Applications*, 229(5) (2015): 403-418.
17. Murray, B.R., Leen, S.B., Semprimoschnig, C.O.A., and Ó Brádaigh, C.M. Rotational moulding of polymer liners for composite overwrapped pressure vessels in cryogenic fuel storage applications. *12th International Symposium on Materials in the Space Environment*. ESA/ESTEC, Noordwijk, The Netherlands, September 24-28, 2012.
18. Kearns, M., Doyle, D., Feerick P.J., Daly, C., and Crawford, R.J. Comparison of cycle times and heating efficiency for direct electrically heated and conventional gas fired rotomoulding machines, *Rotation Magazine*, 13(6) (2004): 18-25.
19. ASTM Standard D1434-82, 2015, "Determining Gas Permeability Characteristics of Plastic Film and Sheeting," ASTM International, West Conshohocken, PA, USA, 2015, DOI: 10.1520/D1434-82R15E01, www.astm.org.
20. Comer, A.J., Ray, D., Obande, W.O., Jones, D., Lyons, J., Rosca, I., O'Higgins, and R.M., McCarthy, M.A.. Mechanical characterisation of carbon fibre-PEEK manufactured by laser-assisted automated-tape-placement and autoclave. *Composites: Part A*, 69 (2105): 10-20.