Feasibility Study Final Report

Feasibility Study (FS) Title: Incorporation of thermoplastic in situ polymerisation in double diaphragm forming

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Partners (include support from Industry): Support received from: Brüggemann (caprolactam ~£1000), Johns Manville (glass fabrics), Tygavac (vac consumables ~£2000), Vac Innovation (vac consumables ~£1000), Engel (technical discussion)

Start date: 1st July 2020 (deferred from 1st March)

End date: 31st March 2021 (after Covid delays)

Executive Summary

Double diaphragm forming (DDF) is a process for producing complex, large preforms for out of autoclave processes. DDF generally requires lower capital investment compared to matched tool forming, but defects such as fibre bridging and fabric wrinkling are more likely to occur. To improve on this, DDF has been combined with a liquid infusion process, where a dry fabric stack held between two flexible membranes (diaphragms) is infused with resin prior to being formed. There are several benefits for doing this:

(1) The resin infusion step is performed with flat fabric plies, simplifying the filling stage without geometrical constraints.
(2) Forming occurs in the presence of a low viscosity liquid, reducing inter-ply friction and diaphragm-fabric friction, improving fabric conformation to the tool
(3) The infusion and forming stages are combined into one process, saving time and consumables

The project fits within the "high rate deposition and rapid processing technologies" priority area of the Hub and addresses both Grand Challenges. It is also complimentary to a number of existing and prior projects funded by CIMComp, including:

1. *Acceleration of monomer transfer moulding using microwaves (2018)*
2. *Manufacturing TP fibre metal laminates by the in situ polymerisation route (2018)*
3. *Active control of the RTM process under uncertainty using fast algorithms (2019)*
4. *Modelling forming of multi-ply preforms (2011);*
5. *Multi-scale modelling to predict defect formation during resin infusion (2011);*

Overall the project delivered on its objectives, but full realisation of the project was limited by travel restrictions due to Covid.

Background

This project specifically considered the use of thermoplastic resin in the Resin Infusion between Double Flexible Tooling (RIDFT) process. Within the 6-months, the RIDFT process was applied to Nylon 6 using in-situ polymerisation, but could be adopted for other thermoplastic resins in the future. Greater understanding of RIDFT and optimisation of its capabilities has the potential to generate a step change in the manufacturing of extremely *large composite parts (+10m): wing spars, boat hulls, train bodies, turbine blades, architectural panels, etc. with relatively minor capital investment costs.*

The project objectives were to, on a small scale:

(a) establish the ability to infuse a flat fabric constrained in a flexible vacuum system (between two diaphragms
(b) determine whether the presence of the resin improves forming of the fabric and
(c) determine the heating requirements to polymerise the resin after forming

This proposed Feasibility Study is applicable to topics within both the 'Deposition/ Conversion' and 'Moulding' themes of the FCMRH Research Landscape Roadmap.

Results/Deliverables/Outcomes

The technical Deliverables (D1 to D4) and success criteria were as follows:

- *D1: Manufacture benchtop diaphragm former, suitable for TP liquid infusion*
- *D2: Produce 2D composite panels by vacuum infusion in rigid tooling (baseline)*
- *D3: Produce 2D composite panels between flexible diaphragms*
- *D4: Produce 3D composite components between diaphragms to at least rigid solid*

Partial success after 6 months – Successful infusion and polymerisation of a flat part between diaphragms to achieve at least a rigid solid state.
Full success after 6 months – Successful infusion, moulding and full polymerisation between diaphragms with improved conformation to tool c.f. control panel, with demonstrated improvement in forming behaviour.

Progress was achieved as follows:

D1 *was to produce a small scale forming system. It was made at a small scale to minimise risks to users and to avoid damage to the existing larger system in early trials. The frame was made taking into account several of the risk factors to try and produce an effective solution and to make it suitable for all processing environments, from fume hood to oven. A three part frame with a vacuum channel provided the double diaphragm section and then a vacuum baseplate with removable hemisphere shape comprised the forming section.*

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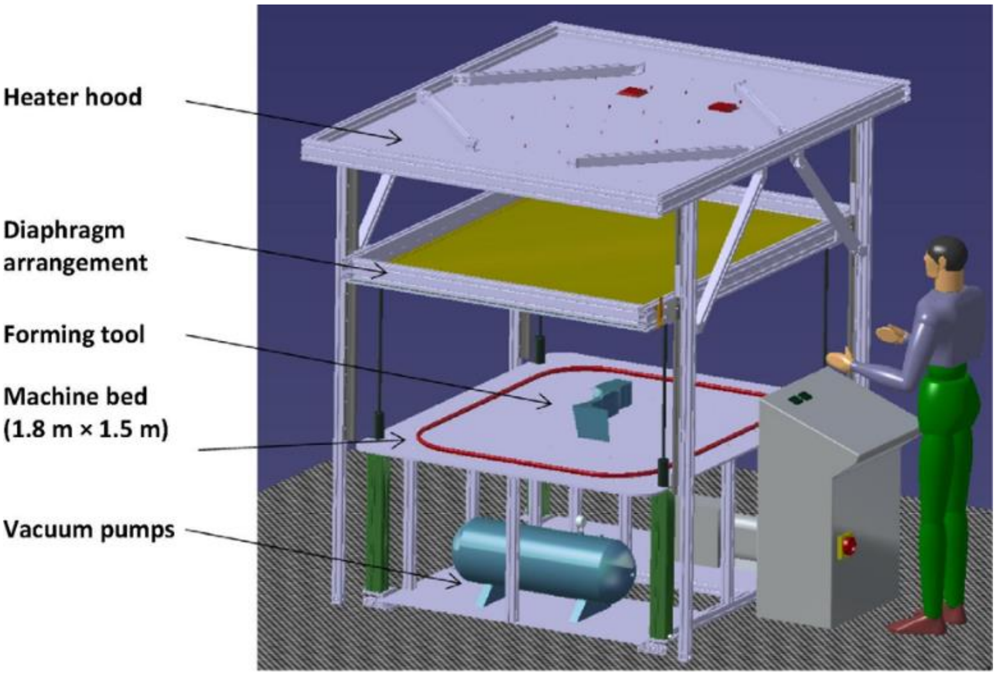
Small scale forming frame with vacuum base and hemisphere former



Larger scale diaphragm former system at UoN

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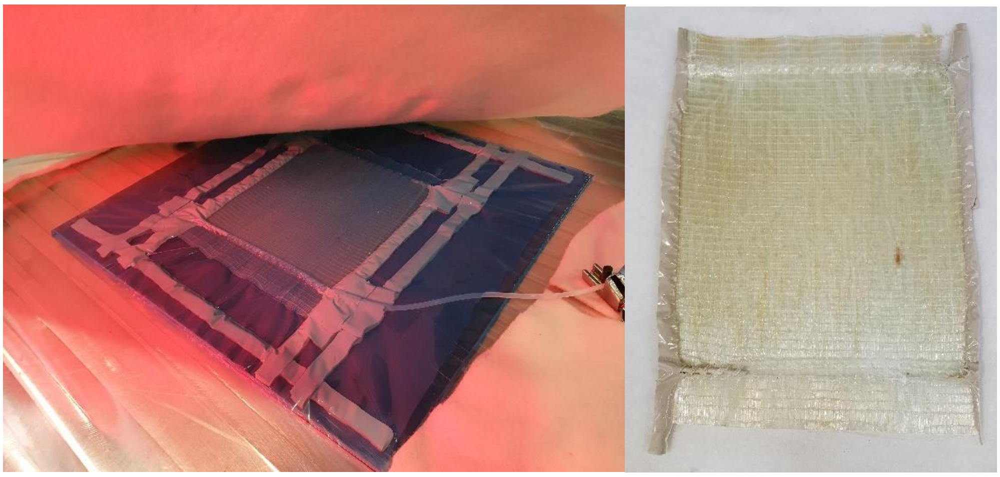
D2 *was to produce a flat panel in rigid tooling using vacuum forming. Some basic laboratory studies were performed to de-risk the process, determining pot life estimation, checking the process would run to completion and most importantly checking the combability of vacuum consumables with the reaction mixture. Most vacuum bagging materials are made of nylon, which is not compatible with the monomer, and so less common materials were considered in consultation with Tygavac and Vac innovation.*

Results identified two fluoropolymer films, normally used as release films but with reasonable strain characteristics (Tygavac A4000, Tygavac Wrightlon), and silicone were all suitable. Tubing was limited to PTFE in high temperature environments and silicone at room temperature. A silicone-based tacky tape was identified, however no suitable breather cloth was available. For the initial trials a slow catalyst was used, to ensure plenty of time to fill and form before polymerisation (30-60 mins). A much faster catalyst is available that would enable faster production (e.g. 2-3 mins).

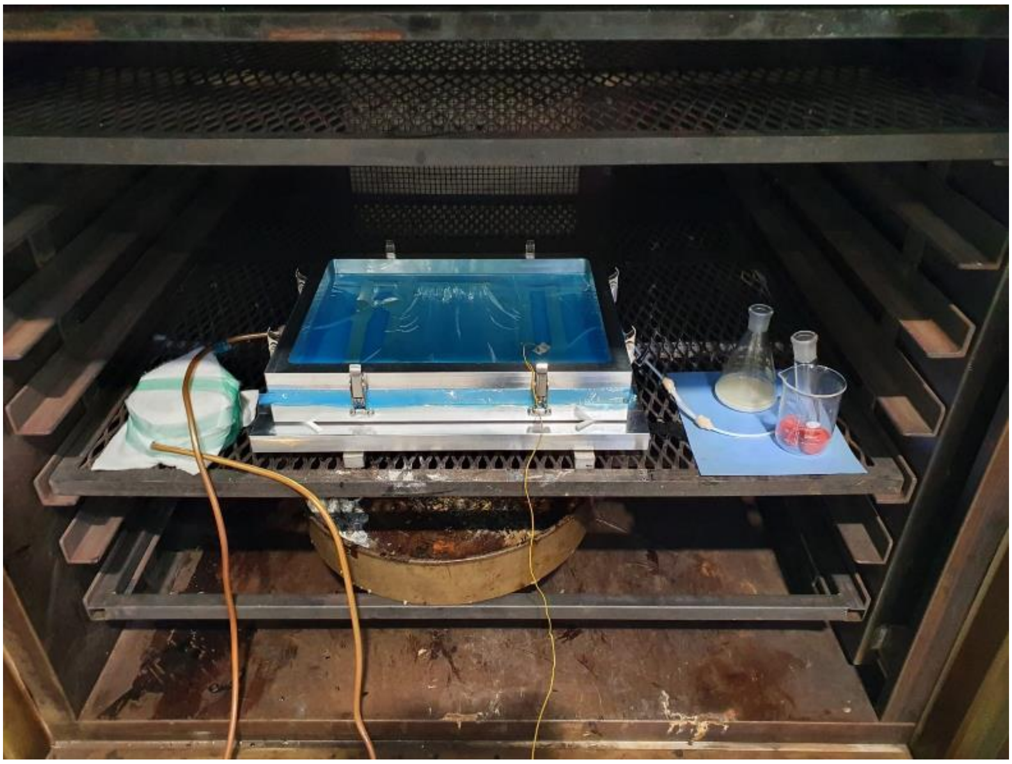
After considerable efforts in developing a suitable infusion protocol using simple resin mixing, flat panels were produced using both glass and carbon reinforcements. The glass and carbon both had specially adapted sizing treatments to suit the APA6 monomer. Infusion was performed between two flexible diaphragms, but was supported by a rigid tool.



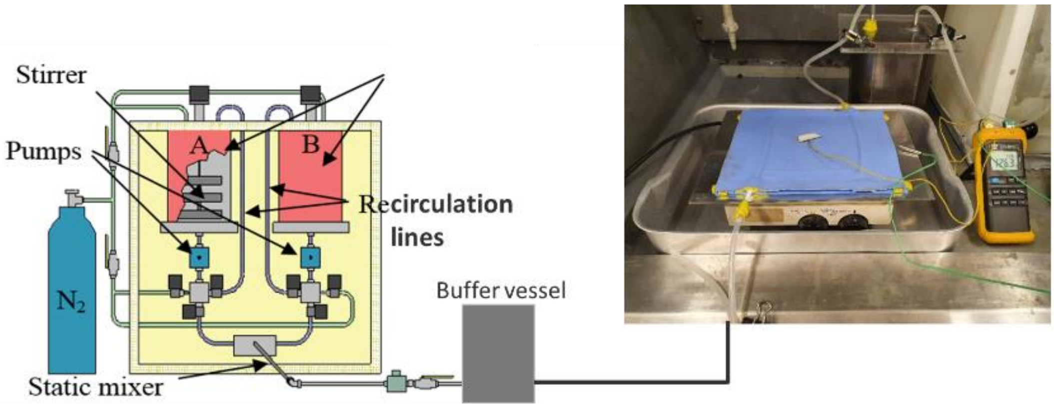
Flat infusion with resulting panel

The heated and purged mixing system at Edinburgh was then incorporated into the process. The system was initially used in it's RTM state, both as a burn in test after recommissioning and to test panel production using the fast and slow catalyst systems. This also ensured that the sizing on the glass, which was originally intended for use with the fast catalyst, was compatible with the slow catalyst. Discussing potential benefits of a mixed catalyst system with Johns Manville will be part of the project review.

After successful RTM production, the mixing system was modified to interface with a vacuum infusion setup, to provide a much improved resin supply to the process. A buffer vessel was used to decouple the pressure of the mixing system from the resin bag infusion. Production of panels was successful through this route.



Vacuum infusion setup in oven at UoN



Schematic of vacuum setup at UoE

To expand potential manufacturing options by decoupling filling from forming, a fill, quench, reheat process was investigated. Infusion was conducted at temperature, before being allowed to cool. The cooled panel was then either reheated immediately to react, or left for a day before reheating. In both cases the reaction appears to have been completed successfully, although degree of conversion is still under investigation. This would potentially allow short term storage of filled frames prior to forming.

***D3** was to produce flat panels in flexible tooling, using the forming frame, with only the vacuum providing rigidity to the system (unsupported diaphragms). Severe racetracking occurred and the original mitigation methods were either impractical or ineffective. Blocking off the vacuum gallery with tape or other material did not prevent the very low* *viscosity resin bleeding through. Modifications to the frame were considered, but were beyond budget in this early study. Instead, perimeter tacky tape was used as a temporary measure to isolate the reinforcement. Through-bag connections were avoided during the infusion stage, to prevent the likelihood of the diaphragm failing during forming due to any stress concentrations.*

While this proved to be entirely suitable for initial trials with an epoxy resin, there were limitations with the in situ polymerisation approach. The infusion was observed to progress rapidly and completely, where excellent wet out was achieved and the resin successfully polymerised to produce approximately 60% fibre volume fraction composites. However, interlaminar consolidation was poor, essentially resulting in a stack of well wet out thermoplastic tapes. This was ascribed to potential loss of vacuum consolidation as a result of a blockage in the vacuum line and/or the action of gravity on the unsupported diaphragms. A number of solutions were investigated, including heated vacuum lines and angled fill, but with mixed success. Ultimately this proved to be a less significant issue when forming (see D4).

Blockage of pipes caused by tacky tape movement

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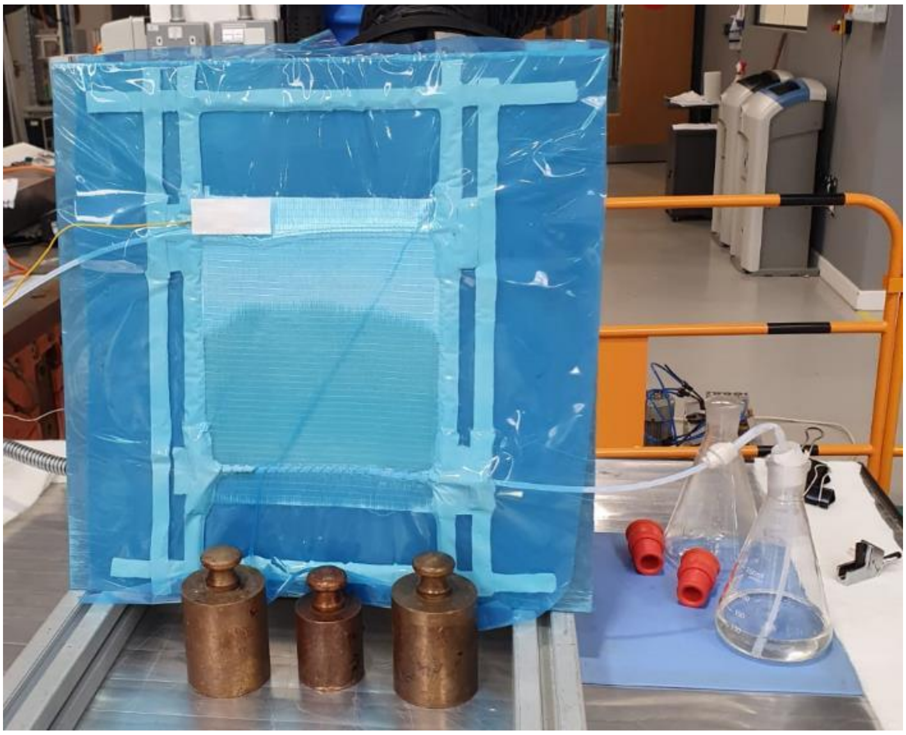
Poor interlaminar consolidation of infused panel



Angled or vertical infusion helped to improve the result

D4 *was to produce formed components using the in situ polymerisation process and ideally to demonstrate the benefits of filling prior to forming. The benefits to forming were first demonstrated with epoxy, where hemispheres produced by first filling a flat reinforcement and then forming (fill-form) achieved better forming results (fewer wrinkles/less bridging) than hemispheres made by forming a reinforcement before infusion (form-fill).*

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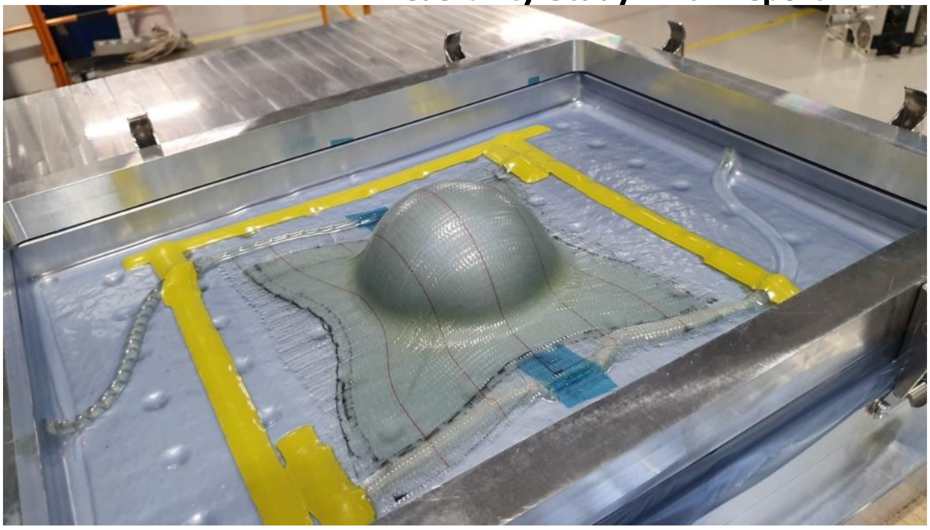
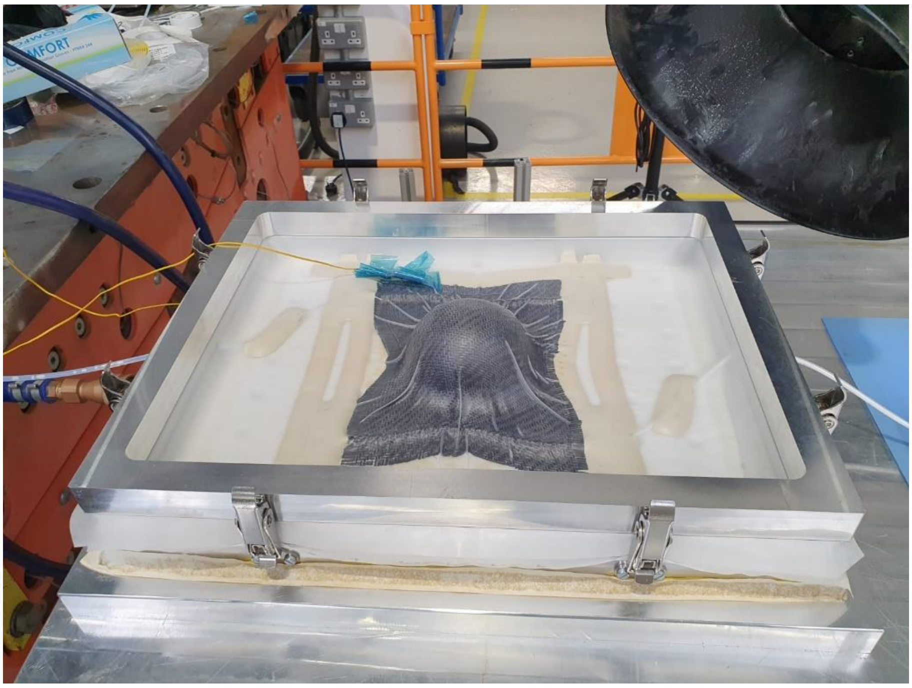


Filled then formed epoxy hemisphere with fewer wrinkles

A successful form-fill experiment has been conducted with carbon fibre and the in-situ polymerisation process. Effectiveness is limited by the consumables and there is an apparent imbalance in pressure acting on the hemisphere. However, the result was well consolidated in the sections that did not experiencing fabric bridging. The Ph.D. student at Nottingham is continuing with this work and additional examples are expected soon. Further refinement of the infusion equipment would improve the result.



Forming with carbon fabric, using a silicone bag

Formed part with dissimilar pressure either side of the former, with significant bridging.

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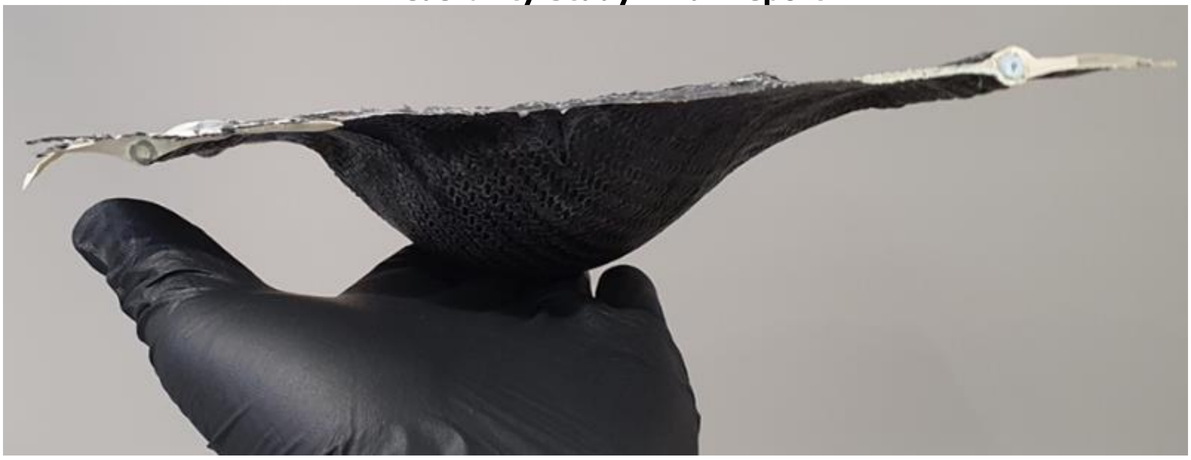

Overall outcome

The final result is somewhere between 'partial' and 'full' success. Most aspects were demonstrated and the process was shown to be feasible at a small scale, but full success cannot be claimed since the infusion mixing system has not been fully combined with the forming frame.

Challenges due to Covid

Covid had a particularly severe impact on the project, causing delays through lockdown and loss of staff time and expertise. In particular the prevention of travel meant that it was not possible to undertake the intended joint activities bringing together the Edinburgh mixing system with the Nottingham forming frame. Shipping one to the other site was considered, but ultimately it was decided that this would not be best use of time as transfer of sufficient required knowledge and expertise virtually was not feasible or efficient. Downtime also had a negative effect on bespoke systems, which needed to be recommissioned after shutdown. The delays did provide a little more design time for the frame and a bit more time for orders to arrive, however supply chain disruption ultimately negated that gain.

Future Direction/Impact

The results of the project are in review with industry partners with a view to scoping out follow on studies. There are continuing activities at both sites at present, but funding is limited. Ideally with the removal of travel restrictions it would be possible to combine the equipment from both sites to demonstrate the process, as was originally intended. As well as process improvements, fundamental questions have been identified in relation to very low viscosity infusion and these would form the basis of an EPSRC supported study (either through a Hub Core Project or a Responsive Mode application). Other opportunities will be explored, potentially in conjunction with the NCC, AMRC or industry. Talks are also taking place with Arkema to discuss the potential of using Elium in this system as well.

Synergy with other Hub projects

There could be potential to work with the Hub project 'Active RTM', however the process would not be immediately transferrable since pressure sensing would be challenging.