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Controlled Preform Consolidation for Manufacturing Difficult Resin System Composites

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Abstract. Novel multifunctional resins and composites present multiple manufacturing challenges that need to be overcome to allow for wider adoption. These challenges include high viscosity, short cure windows, low permeability preforms, and non-standard cure kinetics. Standard liquid composite molding methods (such as resin infusion under flexible tooling [RIFT], resin transfer molding [RTM], or compaction-RTM) are poorly equipped to manufacture these new materials. A new system is presented that combines the abilities of RIFT/RTM/c-RTM while introducing controlled deformation of the preform during infusion to remove flow through the preform. This manufacturing method allows the preform to lie uncompacted, while still under vacuum, during infusion which allows resin to flow unrestricted between plies. Then once infusion has occurred, compaction proceeds to produce the final composite geometry. This method has been successfully implemented to manufacture structural power devices with a biphasic resin system and metal coated carbon aerogel preform, as well as a vitrimer composite with a high-viscosity/short-cure resin infused through high weight carbon fiber preform. The novel and flexible manufacturing parameters of this new system present a low-cost route towards optimizing the manufacture of challenging and novel resin systems, allowing for a faster understanding and implementation of these materials.

Introduction

Resins for modern composite matrices have historically been optimized for mechanical performance and processing requirements. While achieving exceptional properties in these areas, the expanding application of composites necessitates the development of more complex, multifunctional resins to enable greater efficiency in composite structures. These resins can deliver a range of essential functions, such as repairability or energy storage, but they also introduce a new set of processing and manufacturability challenges. They are often more viscous, exhibit narrower processing windows and more demanding handling conditions, and can adversely affect the formability and compressibility of preforms. As a result, substantial modification or reinvention of conventional manufacturing methods is required. In this paper, we examine the requirements for, and potential solutions to, new manufacturing approaches that can be flexibly adapted to a variety of resins, and we present two representative case studies.

Composites with Covalent Adaptive Networks (CANS, aka vitrimers) matrices [1–3] are one such example. Vitrimers are crosslinked polymers, similar to thermosets, that possess side chain chemistry

that disassociates above a set temperature. This allows the material to flow post curing and behave similar to a thermoplastic. Both abilities produce a performative and creep resistant resin that can be repaired [4–6] and reshaped [7,8]. While the processability of this system requires lower heat and pressure than other high-performance thermoplastic systems, vitrimers can be far more viscous and have shorter cure times than comparable thermoset resins. Especially if the reversable side chain chemistry is present in the initial components (rather than synthesized during manufacture). Manufacturing with such systems requires careful consideration to ensure rapid infusion, short flow paths, full preform infusion, achieving good consolidation of infused preforms and high fiber volume fraction, and good surface finish without accumulation of excess resins on ply surface.

Another manufacturing challenge presented in current multifunctional resin research is the development of structural power composites, where the ability to transfer structural load alongside electrical load promises significant weight savings in a wide range of applications. With a specific focus upon structural supercapacitors [9–11], there have been a wide range of novel material developments to improve their electrochemical and mechanical performance in tandem. To allow for the transfer of load and ion transfer concurrently, a biphasic multifunctional resin system has been developed [12]. During cure, this resin undergoes a phase separation to produce a solid, load carrying phase and a liquid, ion carrying phase. This transformation occurs with a complex rheological profile and shortened cure times compared to the base line resin systems it is based on.

The biphasic resin is infused into structural electrodes, which are the carbon woven preforms containing carbon aerogel (CAG) for larger surface area and energy storage capacity. These preforms need to be thin so spread-tow fabric is the most attractive option. CAG occupies the space available between fibers and present much lower permeability. The inclusion of a current collecting film of metal across the entire upper and lower surface of the device further impedes the prospects of through-thickness flow.

The restrictions outlined above produce manufacturing challenges that typical liquid composite molding (LCM) manufacturing processes simply cannot overcome. Resin Infusion under Flexible Tooling (RIFT) [13] is widely used in research settings due to its low cost and flexibility. RIFT relies solely upon a vacuum at the inlet both to produce both the pressure gradient for resin to flow and to apply consolidation pressure. This is typically sufficient for low viscosity commercial resin systems with long cure times but is practically not applicable to a high viscosity vitrimer such as the one explored here. Additionally, the resin distribution mesh creates a rough layer of resin which has considerable thickness compared to the thickness of thin electrodes.

Resin Transfer Molding (RTM) [14], on the other hand, permits to apply positive injection pressure as the preform is constrained by double-sided solid molds. While positive resin pressure gives higher chances for viscous resin to reach the vent, the flow front propagates only in the plane of the preform, which increases the flow length compared to RIFT, while preform is highly compacted, which lowers its permeability. As a result, benefits of positive pressure may be compromised. Inflexibility of RTM also presents difficulties for adjusting it to preforms of various density and compressibility.

Compression-RTM (cRTM) [15] could be an attractive alternative to RIFT and RTM in the context of functional resins. Resin is injected into a mold in a similar manner to RTM but with an open cavity above the preform or with the preform compacted to lower fiber volume fraction that intended. After the injection of the required amount of resin, the upper mold is closed to force the resin through the plies (or drained from the mold), producing the final component geometry. cRTM is capable to take advantage of positive injection pressure, high permeability during infusion, and high level of consolidation achieved in post-infusion compaction. The main limitations of cRTM in the context of structural power applications are the constraints on through-thickness flow imposed by the current collector plies and rather rigid nature of CAG modified carbon fibers preform preventing preform spring back and lowering permeability of the preform during infusion. Infusion trials with a vitrimer resin highlighted limitations in through thickness flow, where almost no vitrimer could be forced through a very limited number of plies of the preform. Complexity and cost of cRTM makes it also rather inconvenient to tune process to the properties of multi-functional resins.

To tackle these challenges a new manufacturing approach is required that can remove the limitation of through thickness flow for novel complex resin and fiber systems. Two trial systems are presented to highlight the abilities of such a novel manufacturing system, the structural power device and vitrimer composites noted above. The proposed manufacturing solution is based around a novel manufacturing system utilizing vacuum induced preform relaxation (VIPR, referred to as vacuum relaxation) [16] to induce the separation of individual preform plies during infusion, as well as independent control of infusion and consolidation pressure. Below we discuss the processing abilities and challenges of this manufacturing route in achieving a cost-effective solution.

Manufacturing System and Materials

Manufacturing system. The novel manufacturing system was designed in house to implement a range of manufacturing capabilities flexibly joining features of RIFT, RTM, cRTM, and VIPR such as positive injection pressure, vacuum at the vent, independent control of consolidation and injection pressures, vacuum relaxation, flexible tooling, and in-tool heating. This has been implemented using a two-chamber design in a rigid tool (Figure 1a). The chambers are separated by a flexible membrane with the bottom half hosting preform, inlets for resin injection under pressure and vacuum vents (Figure 1b). The upper chamber has ports for both applying vacuum or consolidation pressure. The tool is placed in a press to sustain the consolidation and injection pressure, and the flexible membrane serves as sealing as well as pressure transmitting medium.

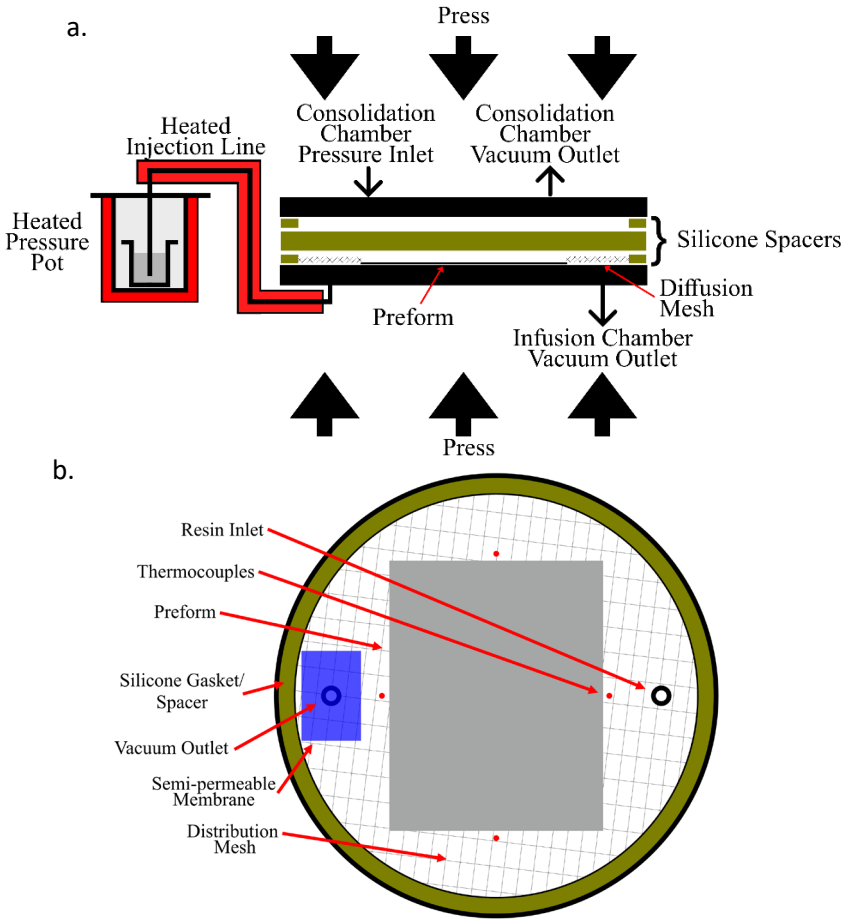

Fig. 1. Infusion system used in this work. Part a presents an overview of the system including all primary components, while part b presents the tool face and how it is set up for infusion. The preform is within the lower chamber of the two-chamber pressurized tool, alongside thermocouples, distribution mesh around (but not on) the preform, and a semi-permeable membrane. Both the upper and lower chambers have a pressure inlet and a vacuum outlet. The lower chamber inlet is connected to a heated pressure pot, via a heated hose, for resin injection. The two-chamber tool is held together using a press to contain infusion and consolidation pressures.

A typical infusion schematic can be seen in Figure 2. When vacuum is applied in both the chambers, the preform will not experience compaction as there is no resulting force upon the silicone membrane. This helps to infuse resin at higher preform permeability and propagate the resin between the plies (Figure 2a). Resin is then infused in-plane driven by the pressure differential between the vent and positive injection pressure. The flow of resin, especially viscous resins, can cause the preform to shift during infusions. While this issue occurred, the resultant force was minimal and small tabs of temperature stable tape (on two opposing corners of the preform) were suitable to prevent movement. The use of a semi-permeable membrane (permeable for gases but not liquid) over the vacuum outlet ensures that resin cannot leave the lower chamber and the full volume is quickly filled, forcing resin into any available spaces such as those between plies (Figure 2b). With the lower chamber filled and the resin delivered between each individual ply, consolidation pressure is then applied to the empty upper chamber. When the upper chamber pressure is greater than the lower chamber, excess resin is pushed back through the injection port and reverse deformation of the preform commences. Extra volume of resin is also accommodated in distribution mesh placed around the preform (Figure 2c). Once all excess resin has been removed the preform forms its final geometry *after infusion has occurred.* The resin-tailored cure cycle then commences by activating the heating elements built within the mold (Figure 2d).

By balancing and sequencing the application of injection and consolidation pressure, various manufacturing routes can be implemented making continuous transition from RIFT to cRTM depending on the processing parameters.

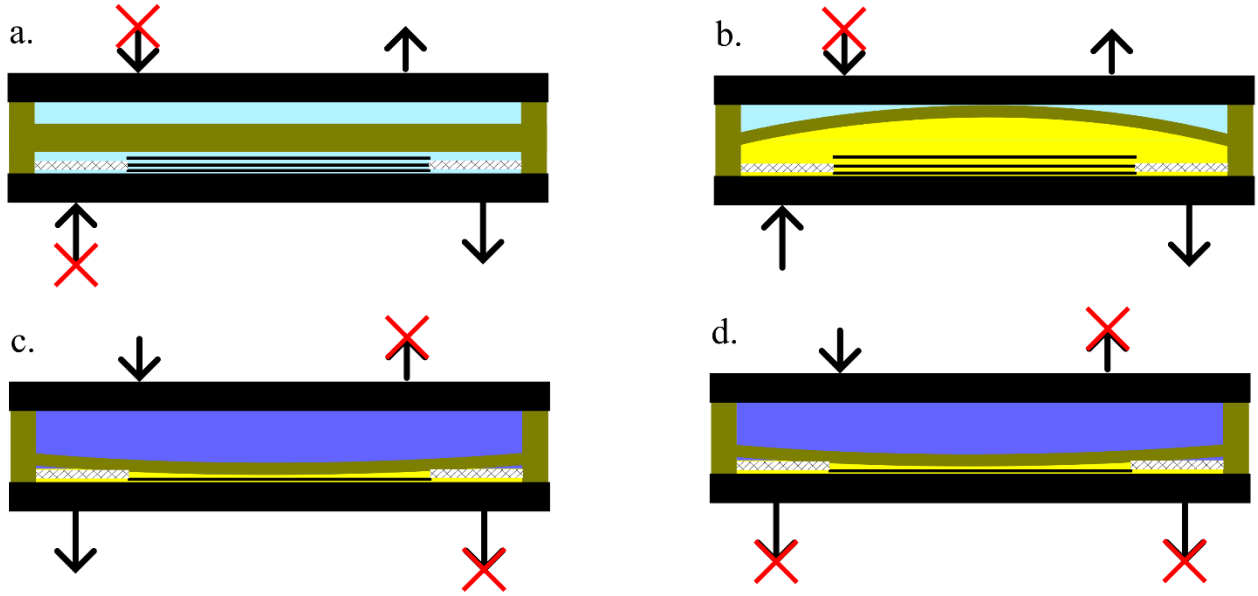

Fig. 2. Composite manufacturing steps in the designed infusion system. a): Both the upper and lower chambers are evacuated to vacuum, with the inlets sealed off. The resulting force upon the membrane is zero, allowing the preform to relax and deform. b): Resin is injected into the lower chamber under pressure. The semi-permeable membrane allows any residual air to escape but stops any resin leaving the lower chamber. As vacuum is still existent in the upper chamber the membrane deforms upwards. c): Once infusion has been completed the vacuum lines, for both top and bottom chambers, are sealed off and the upper chamber is pressurized. Once the upper chamber pressure exceeds that of the lower chamber the membrane is forced down and compaction occurs, forcing excess resin out of the chamber back through the injection port. d): Once all excess resin has left the lower chamber the preform is considered infused and cure progresses.

Structural Supercapacitor Device (SPD). The general design of the structural supercapacitor device is following the methodology developed by Nguen *et al* [10]. The layup consists of two plies of CAG'd spread tow woven carbon fibers (TeXtreme 1134, 12k plain weave 53 gsm fabric, supplied by TeXtreme [17], modified by CAG addition) with a single sheet of a separator between the two plies (1k plain weave Kevlar, 50 gsm was used in this case). Both pieces of CAG modified carbon

fibers have a film of aluminum current collector (3M AL-36FR Tape, purchased from Mouser Electronics [18]) glued, with conductive adhesive, to the outside faces of the laminate. The total laminate thickness is 0.2 mm, with a maximum size of 210x148 mm (a5 paper size). Trial infusions were of this size but samples for electrochemical testing were 40x40 mm, following outlined protocols of Greenhalgh *et al* [19]. No flow medium was placed above or below the preform, but to aid with excess resin removal during compaction the rest of the tool face was covered in distribution mesh (Figure 1b).

The structural electrolyte (SE) infusion resin, modified from the original design by Wengdong *et al* [12], consisted of Huntsman Tactix 742 epoxy (RD4, supplied by Huntsman Resin [20]) and isophorodiamine hardener (iPDA, purchased from Merck [21]) mixed at a 1:4 wt. ratio respectively. The epoxy was heated to 65 °C and 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIM TFSI, >99.5%, purchased from Iolitec [22]), aka ionic liquid (IL) was added at 40 volume% to the reaction mixture and stirred by hand. Once cooled, the hardener was added, mixed by hand, and then formed solution was used to infuse at room temperature with 6 bar of injection pressure while the preform was held under vacuum relaxation. After the chamber was filled with SE the injection pressure was lowered to 2 bar and the upper chamber was slowly pressurized to 6 bar. The infusion was considered complete once excess resin stopped flowing out of the chamber, and the cure cycle followed. The resin cure cycle was as follows: 16 hours at 25 °C, 1 hour at 60 °C, 2 hours at 80 °C, 2 hours at 120 °C. At room temperature the resulting resin has a viscosity of ~20 Pa.s. Phase separation starts to occur around 60 minutes after mixing, with a resin pot life of about 100 minutes.

Vitrimer Composites (VC). Four plies of NCF unidirectional 50k, 600 gsm carbon fiber fabric (with tricot synthetic stitching, layup $[0, 90]_s$) was used as the infusion preform. This was chosen both for the challenge of the large tow size, as well as the heavy material weight acting against preform relaxation. Infusion occurred with a flow/deformation guide within the infusion chamber to aid both the deformation of the preform and to reduce the internal volume of the mold. Mallinda VHM epoxy and hardener [23] was used as the infusion resin. Both parts of the resin were heated to 85 °C before being mixed in the manufacturers recommended ratio of 1:1.5 of epoxy to hardener respectively. The pressurized injection pot and infusion line were heated to 80 °C (equipment maximum) and the tool heated to 85 °C. The mixed resin has a viscosity of ~6 Pa.s, with a pot life of 150 minutes. The resin was injected at 6 bar of pressure into the vacuum relaxed lower chamber and held until the volume was filled. The injection pressure was then lowered to 2 bar and 6.5 bar of consolidation force was slowly applied. The infusion was considered complete when excess resin was no longer flowing back out of the infusion chamber. Resin cure then progressed with 1 hour at 85 °C.

Results and Discussion

Optimization of the infusion system was required before high quality infusions were achieved. Parameters such as infusion time, infusion pressure, tool heating, distribution mesh use and lay-up, infusion chamber volume, consolidation chamber volume, chamber volume ratios, and consolidation pressure were all explored during initial trials. Chamber volume, and the specific ratio between the upper and lower chamber, was discovered to be key to ensuring optimum infusion. If the upper chamber was too large for example, then the membrane would lift significantly during vacuum relaxation. The resulting excessive volume in the infusion chamber either (i) appears difficult to drain in post-infusion consolidation particularly when resin quickly gels, which leaves substantial resin rich domains on the composites surface (Figure 3a), or (ii) if the chamber is not fully flooded, accumulates in the distribution mesh surrounding preform but losing pressure gradient at the flow front to spread over the entire area leaving the preform dry (Figure 3b). In addition, the large volume would require an excessive amount of resin to fill the volume, making infusions both expensive and wasteful. Meanwhile, if the chamber volume is too small, vacuum relaxation does not permit the accumulation of sufficient amount of resin. The cavity thickness was varied in sequential trials and different optimum thicknesses were found for the two infusions explored here. All infusions required a lower internal height equal to, or slightly greater than, their final product thickness. However, the upper chamber height was dependent on the height of the dry fiber preform when unrestrained. The VC

(with a 4 mm final product thickness) required an upper chamber height of roughly 3 mm, while the SPD infusions (with 0.2 mm final product thickness) required an upper chamber height of less than 1 mm. These values are to be taken alongside the lower chamber height. For example, the VC composite infusion had a lower chamber height of 4 mm, while an upper chamber height of 3 mm, combined to 7 mm. This 7 mm is the height of the preform when unrestricted during layup, therefore in its most uncompacted state and providing least resin flow restriction. Ensuring the combined chamber heights allow for full fiber relaxation, without producing too large a volume (making it difficult to remove excess resin after infusion, is key to producing high quality infusions.

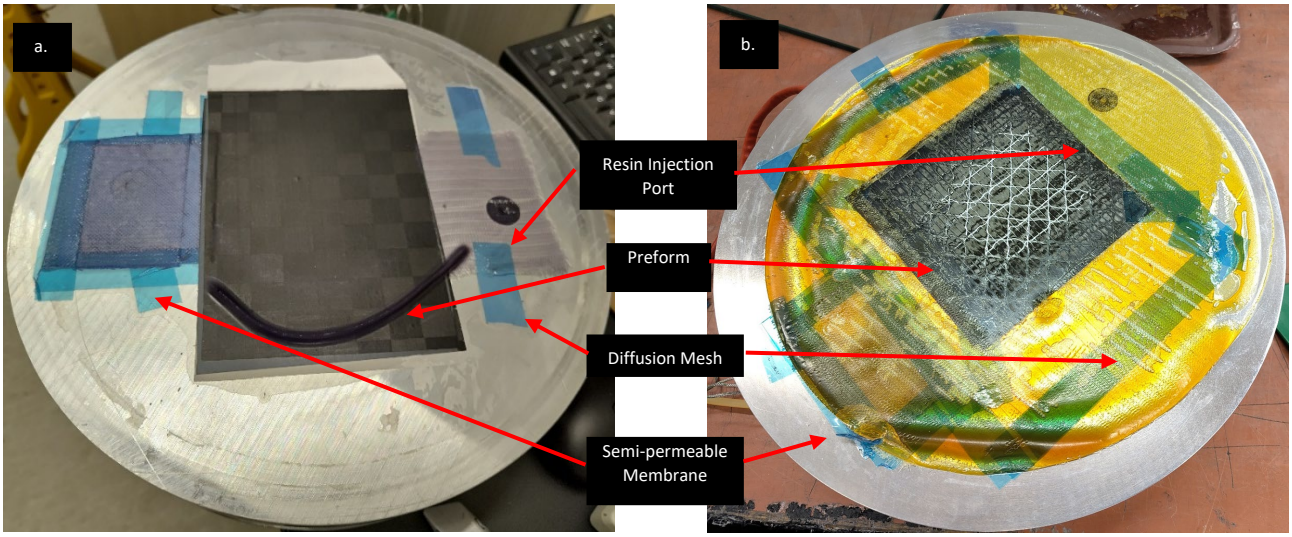

Fig. 3. a): Failed infusion of SPD device using a monofunctional version of the SE resin, where excess upper chamber volume caused a trapped vein of resin during consolidation. Purple color was added to the resin in early infusions to aid in resin identification and flow understanding, along with minial use of flow distribution mesh. b): Failed infusion of preform with vitrimer resin. The internal volume was too small to allow for adequate vacuum relaxation. Of note is the white dry fiber stitching still visible in the center of the laminate that has been removed around the edges of the preform. When comparing to Figure 1b, the thermocouples are not visible, the vacuum outlet port is hidden under the semi-permeable membrane, and the silicone gasket has been removed.

Early infusion trials, such as one shown in Figure 3b, also tended to only partially infuse the preform. This was due to the internal height within the infusion and vacuum relaxation chambers being too small. Interestingly, where infusion has occurred the white stitching yarns of the preform have been removed (thought to be dissolved). An optical image of the center of a later, successfully infused VC laminate is shown in Figure 4. It still has the stitching present (darker fibers), while the surface of the laminate has no stitching present like in Figure 3b. While the fibers are thought to be dissolved in the resin, there appears to be a limiting factor with the designed infusion method that is limiting the amount of stitching that can be dissolved in the center of laminates. To fully understand this process, further understanding of the flow mechanisms and characteristics within the preform during vacuum relaxation is necessary. The SPD infusions may also have similar factors occurring during infusion but they are not noticed due to the lack of an indicator such as stitching. If these mechanisms can be better understood, then the removal of the stitching may aid in compaction and improving future infusions.

Additional work was also undertaken on the infusion aids within the chamber. Initial runs aimed to use no flow medium at all. However, it was quickly discovered that resin ended up being trapped between the tool and the membrane during compaction, leading to large excess resin regions across both the tool and part. Thie necessitated the inclusion of distribution mesh across the bare tool face, but not on the preform itself, to allow for excess resin to flow out the chamber during compaction. Many infusion trials were undertaken to optimize these parameters to improve infusions, with

emphasis placed on minimizing resin use (influenced by the high cost of the IL used in this work) alongside high part quality with minimal voids.

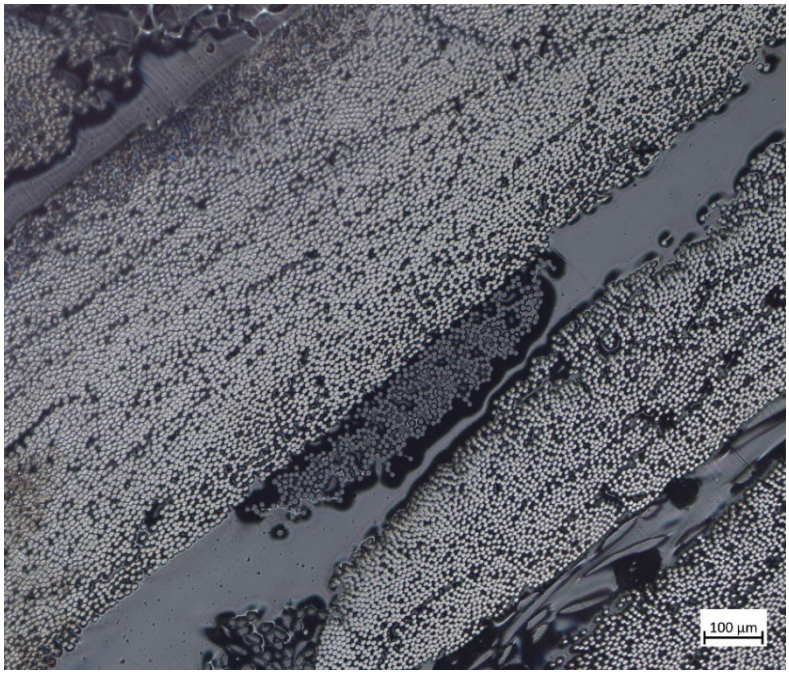

Fig. 4. Optical microscope image of the infused vitrimer samples. The white fibers are the carbon fibers of the laminate, while the darker fibers are the NCF fabric stitching.

Utilization of the preform deformation during the SPD manufacture produced a uniform laminate (Figure 5), with almost no voids. The RD4 SE microstructure in SPD was even and uniform across the entire device. Figure 6 shows an SEM image of the infused separator, with the gaps between the Kelvar fully infused with RD4 SE resin.

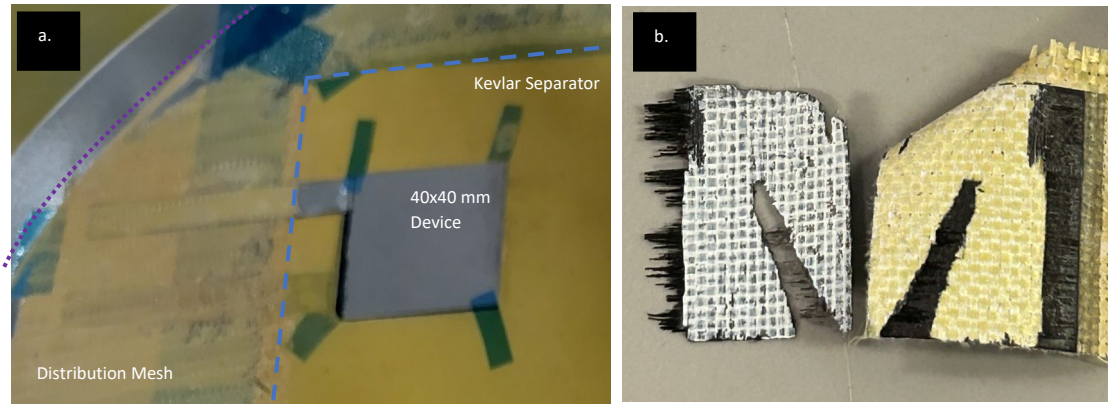

Fig. 5. The infused structural supercapacitor 40x40 mm test device (a.) and the same device dissected for SEM imaging (b.). The current collector is the metallic top layer seen in image a with electrical connection tabs running off the left side of the device for both positive and negative connections, with the black CAG'd carbon fiber and yellow Kevlar electrical separator (outlined with blue dashes in a) can be seen in both images. The distribution mesh is outlined in purple dots in image a. In image b, the infused RD4 SE is the white seen on the peeled carbon fiber.

Infusion of the vitrimer material underwent similar optimization before successful infusions were possible, where upon full infusion through all plies and into the center of the 50k tows (Figure 4) was possible. Vacuum relaxation ensured that the preform had no inter-ply flow restrictions (along the x- and y-axis, between plies). This greatly aided the infusion by reducing the resin flow through thickness to only half of a ply, compared to the full z-thickness in typical RIFT/c-RTM infusion. While the compaction of the laminate does appear limited due to the presence of resin-rich

interlaminar regions, this is thought to be due to the inclusion of the flow/deformation guide and the interlaminar stitching not fully being removed during infusion. The flow/deformation guide greatly reduced the volume within the lower infusion chamber by occupying all space that was not the inlet port, outlet port, and the preform between them. However, this silicone sheet possibly supported the chamber separating membrane during compaction and reduced the pressure applied directly to the preform. Overall the infusion was still far more successful than any other VC manufacturing trialed and presents a viable route for vitrimer composite manufacturing with short cure, high viscosity resins.

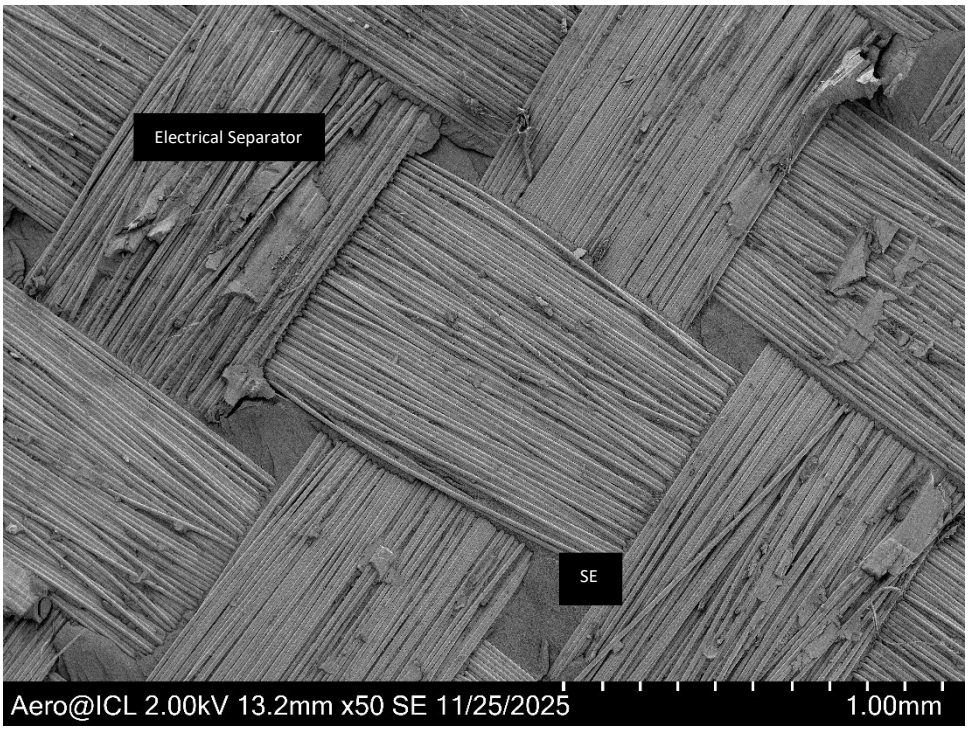

Fig. 6. SEM image of the electrical separator infused with structural electrolyte (SE), separated after infusion.

Conclusion

The successful manufacture of challenging multifunctional resin systems has been achieved utilizing designed preform deformation within a novel manufacturing solution, showcased through the production of a structural supercapacitor device and a vitrimer composite. The novel solutions has allowed for the infusion around low-permeability preforms, reduced required resin flow paths to only half a ply, and enabled a cost-effective solution to trialing novel material systems. The incorporation of elements from a range of typical infusion techniques (RIFT, RTM, cRTM) and the introduction of VIPR has produced a working hybrid that enables manufacture that none of the typical methods could achieve themselves. While the flexibility and low cost of the designed system is beneficial, the higher use of resin can be limiting. The entire chamber volume requires being filled with resin before compaction. The explored examples here are relatively thin, but even the 4 mm thick vitrimer composite required a volume reducing guide to ensure the full chamber volume was filled, and compacted, before gel point was reached. Overall though, the designed system appears to allow for rapid optimization of an infusion process, enabling key infusion parameters to be explored with greater flexibility than traditional pressure infusion systems (RTM and cRTM), while not sacrificing much ease of use when compared to typical RIFT infusion. Going forward, further experimental validation on new complex resin systems will enable far quicker optimization of manufacturing conditions with the added advantage of vacuum relaxation to allow for the controlled deformation of preforms to reduce flow restrictions throughout the preform.

Acknowledgments

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