



# A REVIEW OF OUT-OF-AUTOCLAVE PREPREGS - MATERIAL PROPERTIES, PROCESS PHENOMENA AND MANUFACTURING CONSIDERATIONS

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**Abstract:** Out-of-autoclave (OoA) prepreg materials and methods have gained acceptance over the past decade because of the ability to produce autoclave-quality components under vacuum-bag-only (VBO) cure. To achieve low porosity and tight dimensional tolerances, VBO prepregs rely on specific microstructural features and processing techniques. Furthermore, successful cure is contingent upon appropriate material property and process parameter selection. In this article, we review the existing literature on VBO prepreg processing to summarize and synthesize knowledge on these issues. First, the context, development, and defining properties of VBO prepregs are presented. The key processing phenomena and the influence on quality are subsequently described. Finally, cost and environmental performance are considered. Throughout, we highlight key considerations for VBO prepreg processing and identify areas where further study is required.

Key words: A-Short-Fibre Composites, C-Finite Element Analysis (FEA), C-Modelling, Mean-field homogenization



## 1. Introduction

Advanced composite materials based on carbon fiber-reinforced thermoset polymers have become common in primary aerospace structures, as well as high performance sporting goods, and marine and wind energy structures. As these composite parts grow in number, size and complexity, the need for faster, more cost-effective and more versatile manufacturing comes into conflict with the limitations of traditional processing methods.

Most high performance structural composites for aerospace applications begin as layers of prepreg, or carbon fiber beds pre-impregnated with a catalyzed but uncured resin [1]. Traditionally, prepreg layers are stacked on a tool to form a laminate, enclosed in a vacuum bag assembly, and placed in an autoclave (pressurized oven). The autoclave temperature is then raised, partial or full vacuum is drawn in the bag, and the vessel is pressurized. The consolidation pressure differential compresses the fiber bed, conforms the laminate to the shape of the tool and, in some cases, forces out excess resin. The applied pressure also suppresses porosity, the main manufacturing defect in prepreg-based parts, by driving resin into dry areas and collapsing bubbles of entrapped air and/or cure-generated volatiles. Concurrently, the elevated temperature reduces the resin viscosity, allowing resin to flow and wet the reinforcement before curing into a stiff, strong solid.

Autoclave processing is robust and well-understood, having benefited from significant research and experience gained from widespread industrial use, and remains a benchmark for competing processes [1, 2]. However, autoclaves involve significant costs for acquisition, operation, and tooling, particularly for large parts. Autoclaves also impose a relatively inflexible manufacturing environment, in which potential part designs are constrained by available vessel sizes, production

rates are restricted by scheduling, large autoclaves must sometimes be used inefficiently for small parts. Please cite this article as: Centea T, Grunenfelder LK, Nutt SR. **A review of out-of-autoclave prepreps - material properties, process phenomena and manufacturing considerations.** *Compos Part A Appl Sci Manuf* [Internet]. 2014; 70:132–154. Available from: <http://www.sciencedirect.com/science/article/pii/S1359835X14003108>



parts, and subcontractor options are limited. Given the predicted market growth for composites and the aforementioned limitations of autoclave processing, out-of-autoclave (OoA) manufacturing techniques, particularly those that yield autoclave-quality parts, are required to meet future demand.

Recently, a new generation of out-of-autoclave prepregs has been introduced, and experience with these prepregs has demonstrated that it is possible to produce autoclave-quality parts using vacuum bag-only (VBO) consolidation. By avoiding the use of autoclaves, such materials significantly reduce acquisition and operating costs, and are compatible with a diverse range of lower-cost cure set-ups, including conventional ovens, heating blankets, and heated tools. In addition, the lower cure pressure supplied during VBO cure can eliminate autoclave-induced defects such as honeycomb core crush, allowing the use of lighter (and less expensive) cores. Questions remain, however, as to the ability to produce void-free primary structural parts out-of-autoclave and the true economic benefits of VBO cure. In 2011, High Performance Composites magazine ran an article entitled “Out-of-autoclave Prepregs: Hype or Revolution?” [3]. Here, we revisit this question by considering over two decades of publications on OoA processing, compiling, for the first time, the relevant literature on the topic.

## 1.1 Background

Initial work on out-of-autoclave prepregs was performed by prepreg manufacturers and their industrial partners. The development of VBO prepregs, therefore, is documented in a decade-long series of patents and conference proceedings [4–15]. These publications discuss the context and rationale behind VBO prepregs and describe general characteristics. Research carried out in an industrial context is often only selectively published due to intellectual property considerations.

However, in recent years, research universities throughout the world have undertaken efforts aimed

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at a fundamental understanding of VBO cure. There is, at this point, sufficient published data on VBO cure to warrant a comprehensive review of the topic, relevant to both academia and industry.

Early-generation VBO prepregs were designed for low-temperature initial cure ( $\sim 60^{\circ}\text{C}$ ), followed by high-temperature post-cure, and intended for low production runs or load-limited structures [4,5,7]. The main advantages of these materials were the ability to use lower cost tooling, combined with an increase in dimensional accuracy because of reduced tool thermal expansion. However, these benefits were outweighed by three major drawbacks: (1) relatively high porosity resulting from low applied pressure or inconsistent resin bleed, particularly for high fiber volume fraction reinforcements [4,8]; (2) out-times, or allowable room temperature storage times, of only about a week [4,8,16]; and (3) relatively low mechanical performance, particularly in terms of toughness [4,16]. Resin formulators noted, however, that in typical practice, such systems were cured in the  $80^{\circ}\text{C}$  to  $100^{\circ}\text{C}$  temperature range [4]. This hotter, wider process window, coupled with developments in resin chemistry and an increasing understanding of optimal matrix properties, enabled the development of a new generation of VBO resins. When properly integrated into appropriate fiber bed architectures and correctly processed, these materials were competitive with autoclave systems on multiple fronts, including porosity [5,6,11], mechanical performance [5,10], and out-time [11]. Several such resin systems are shown in Table 1, most of which can be coupled with a range of reinforcements, including woven carbon and glass fiber fabrics and unidirectional (UD) tapes. While a diverse range of VBO prepregs are commercially available, commonalities exist across all low-pressure curing prepregs.



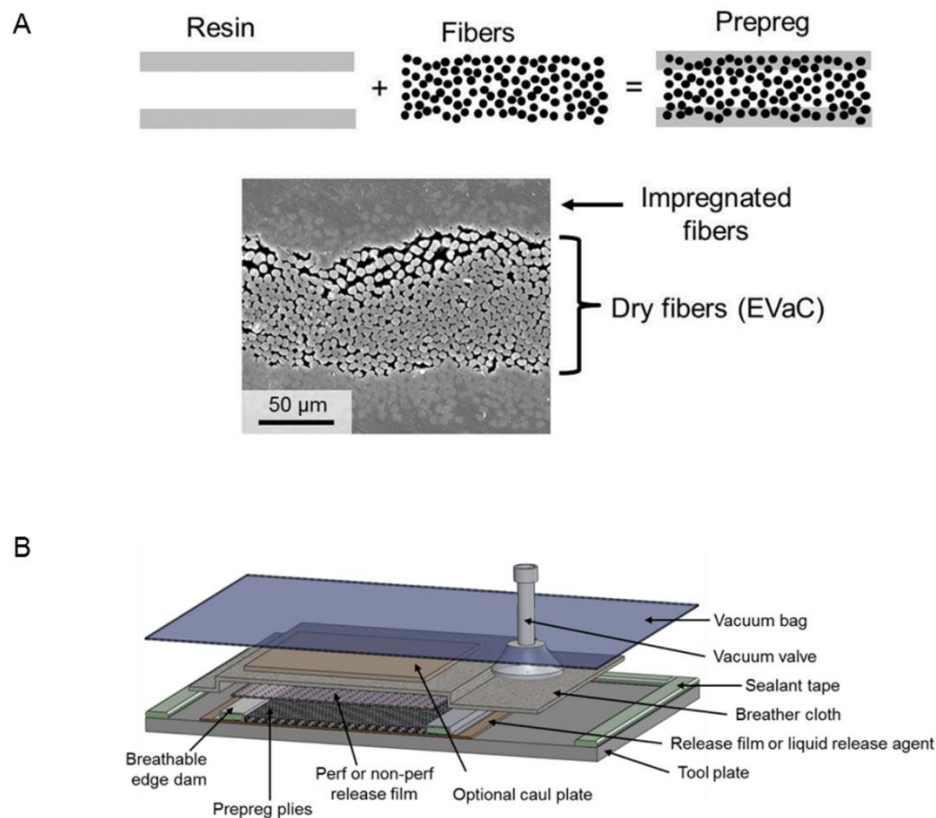
**Table 1:** Current-generation aerospace grade OOA/VBO prepreg resin systems.

MANUFACTURER	RESIN FAMILY	RESIN TYPE	DESCRIPTION
<b>ACG (now Cytec)</b>	MTM44-1	Epoxy	Medium temperature molding (MTM) toughened epoxy. Qualified by Airbus for secondary and tertiary structure
	MTM45-1	Epoxy	Lower temperature cure system optimized for compression performance
	MTM45-1FR	Epoxy	Variant of MTM45-1 optimized for flame retardation
	MTM47-1	Epoxy	Variant of MTM45-1 optimized for hot/wet notched performance up to 130°C
<b>Cytec</b>	Cycom 5320	Epoxy	Toughened epoxy designed for primary structure application
	Cycom 5320-1	Epoxy	Variation on 5320 system, formulated for increased out-life.
<b>Gurit</b>	Sprint ST94	Epoxy	Single-sided moulding prepreg for parts requiring resistance to impact damage and microcracking
<b>Hexcel</b>	Hexply M56	Epoxy	High performance VBO epoxy system
<b>Toray</b>	2510	Epoxy	Formulated to meet the requirements of general aviation primary structure
<b>Tencate</b>	BT250E	Epoxy	Standard VBO system used in Cirrus aircraft and unmanned vehicles. Variations for fatigue and fracture resistance for helicopter rotor blades
	TC250	Epoxy	Second generation VBO system with increased toughness and higher service temperatures
	TC275	Epoxy	Third generation system with greater inspectability, resistance to hot/wet conditioning and curable at 135° C
	TC350-1	Epoxy	Third generation system with increased out-life (45+ days), high toughness, and ability to cure at 135° C with 177° required post cure
	TC420	Cyanate ester	High temperature system (service temperatures up to 315° C)
	TC800 BMI+	Bismaleimide	High-temperature, toughened BMI prepreg formulated for cure out-of-autoclave
<b>Henkel</b>	Loctite BZ	Benzoxazine	VBO prepreg based on a blended epoxy-benzoxazine resin formulation

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Repecka and Boyd [8] and Ridgard [5] outline several features commonly encountered in VBO preregs, and emphasize important layup, bagging and cure characteristics. The key requirement for low porosity VBO-cured parts is the removal of air entrapped during lay-up. To this effect, VBO preregs are “breathable,” featuring partially impregnated microstructures consisting of both dry and resin-rich areas (Figure 1A, from [17]). The dry areas, sometimes denoted as “engineered vacuum channels” or “EVaCs,” form a relatively permeable vascular network that allows gas migration towards the laminate boundaries in early processing. When the temperature is increased, resin flows into and infiltrates these channels, leading in principle to a void-free part.



*Figure 1: (A) Schematic and SEM micrograph of air evacuation channels in VBO prepreg; (B) VBO layup schematic. Reproduced with permission [17]. Elsevier, 2013.*

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To allow gases to escape from the prepreg into the breather, vacuum bag assemblies must include permeable boundaries that connect the laminate to the breather cloth without allowing excessive resin bleed. For in-plane gas evacuation, these boundaries take the form of dry fiberglass strands, cork or other “edge breathing” dams placed around the laminate perimeter. For through-thickness air evacuation, perforated release films or peel plies can be used to separate the laminate from the breather. A common VBO lay-up, with appropriate consumables identified, is presented in Figure 1B. For the interested reader, a detailed description of lay-up and bagging procedures for small VBO laminates was reported by Lucas et al [18].

The cure cycles used to process VBO prepregs also feature notable differences from those used for autoclave materials. First, because air evacuation and consolidation pressure are essential, the bag vacuum quality must be high throughout cure. A minimum vacuum gauge reading of 28 in Hg, corresponding to a maximum bag pressure of approximately 6500 Pa, is generally recommended, and the importance of a leak-proof bag is emphasized. Second, a room-temperature vacuum hold ranging from a few hours for small parts to more than 16 h for large ones is required prior to cure to evacuate gases entrapped during layup. Third, OoA prepreg resin systems are designed for initial cure (impregnation, gelation and vitrification) between 80°C to 120°C, and subsequent in-bag or free-standing post-cures at up to 177°C are used to complete cross-linking and maximize thermochemical properties.

In summary, for successful cure, VBO prepregs rely on specific material and prepreg properties and appropriate process parameter selections. Furthermore, in the absence of a high-pressure safeguard, they are likely to be sensitive to unintended deviations from ideal conditions. In this context, properties and processing must be thoroughly understood.

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## 1.2 Objectives, Structure and Scope

In this review, we assemble, summarize and synthesize published literature on VBO prepreg properties and processing with three primary aims: (1) to compile and organize existing knowledge, which is remarkably disparate in both source and location, (2) to highlight key material and process features, which are more complex than for traditional autoclave prepreps, and (3) to contrast the VBO and autoclave methods in hopes of facilitating transitions between the two.

First, we describe the prepreg, fiber bed, and resin properties. Then, we review literature on several aspects of processing, including air evacuation, flow/compaction and voids, geometric complexity, and honeycomb structures. Third, we survey cost and environmental performance studies. Finally, we synthesize and discuss the salient trends and major themes of the literature, and draw conclusions.

This review is intentionally focused on late-generation VBO prepreps intended for aerospace (or similar) applications, because the shared constituent properties and microstructural features of these systems enable them to compete with high performance autoclave materials. Therefore, studies on the non-autoclave cure of autoclave prepreps as well as on older or lower-performing VBO prepreps (such as those intended mainly for automotive or marine structures) are discussed only when relevant. Furthermore, literature pertaining to other non-autoclave methods (such as vacuum-assisted resin infusion, resin transfer molding, or Quickstep™) is omitted.





## 2. VBO Prepreg Properties

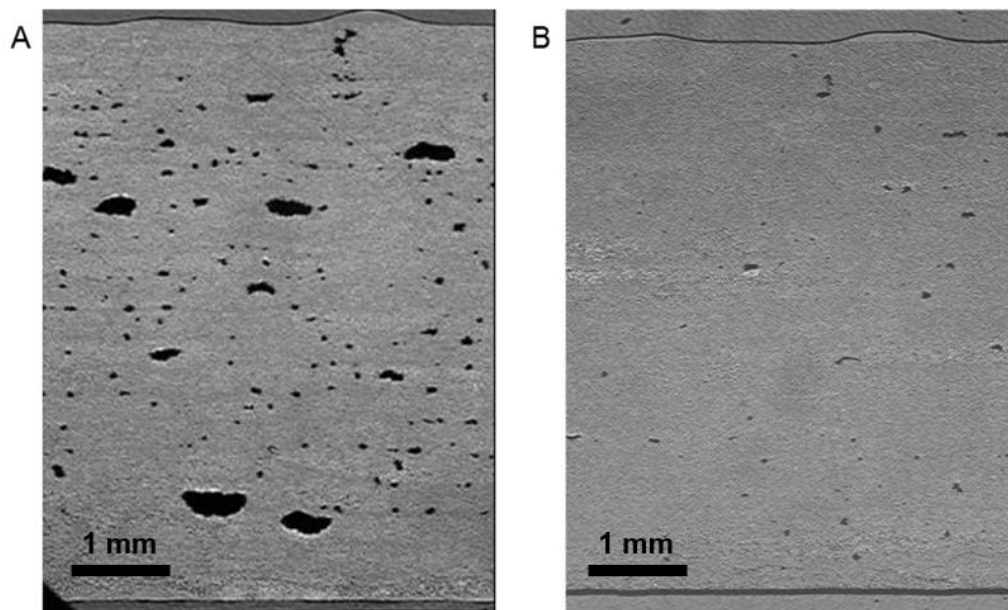
### 2.1 Prepreg

The key to the production of void-free parts using out-of-autoclave methods is, counterintuitively, the introduction of open-cell porosity into the prepreg itself, through partial impregnation. The link between prepreg impregnation and porosity was first established by Thorfinnson and Biermann [19,20] in the 1980s. At the time, thick autoclave-cured parts required time-consuming intermittent vacuum compaction (or debulking) and sometimes inconsistent resin bleed to achieve low porosity. While attempting to develop more time-efficient strategies for autoclave cure, the authors discovered that reducing the degree of impregnation (DI) of the prepreg led to lower void contents, even in the presence of typically challenging features such as ply drop-offs [19]. This result indicated that dry areas within each prepreg ply can allow the evacuation of entrapped air, vaporized moisture, and other volatiles, before being impregnated with surrounding resin. Subsequently [20], the authors considered the DI in greater detail. First, they formally defined it as the ratio of resin-saturated interstitial volume to total interstitial volume in the prepreg, and proposed a mercury porosimetry technique for measuring it. Then, they provided experimental data correlating DI to panel quality. For the same resin and fiber materials, a panel made from prepreg with a DI of 60% was void-free, while one based on a DI of 93% featured significant interlaminar voids. Most interestingly, an intermediate DI of 82% was reported to produce both high- and low-porosity panels, depending on the vacuum quality and room temperature vacuum hold time, indicating that other material properties and process parameters may be influential. The partially



impregnated preregs developed by Thorfinnson and Biermann were referred to as thick laminate preregs, or TLPs.

Over a decade after this initial publication, the first paper introducing vacuum bag-only preregs was published by Repecka and Boyd [8]. In this work, TLP technology was utilized in the development of Cytec Engineered Materials' (now Cytec Industries) first generation VBO preregs based on low-temperature, no-bleed resin systems (5215 and 754). As a demonstration, two panels were produced in this study using identical resin systems and fiber reinforcements, varying only the degree of prepreg impregnation. Both panels were cured using vacuum bag-only techniques. The traditional, fully impregnated prepreg led to a laminate void content exceeding 5%, while the TLP design resulted in a nearly void-free panel (Figure 2). Other prepreg producers have utilized similar approaches in the development of VBO prepreg systems.



*Figure 2: Parts cured out-of-autoclave using (A) conventional prepreg and (B) thick laminate prepreg, or TLP, a precursor to VBO preregs. Adapted and reproduced with permission [8]. Society for the Advancement of Material and Process Engineering (SAMPE), 2002*

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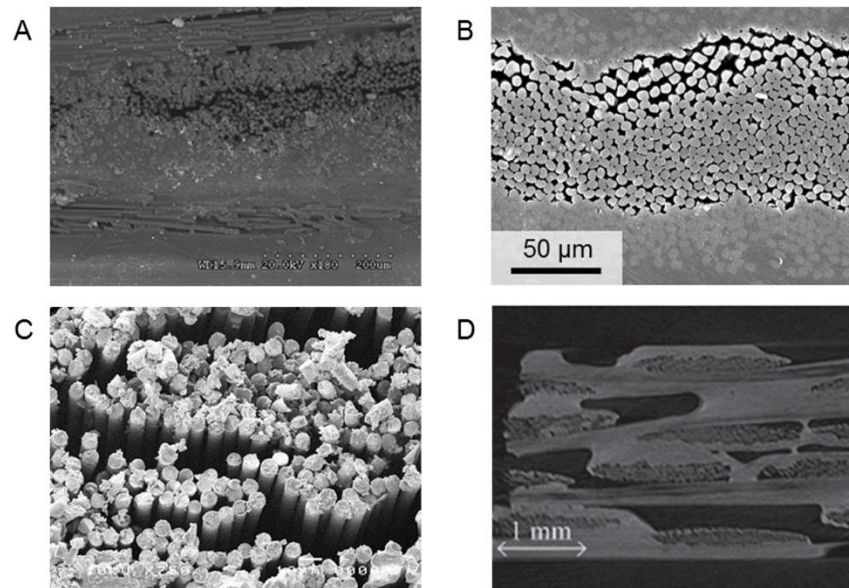


Utilizing a slightly different design, Gurit developed a VBO prepreg class known as SPRINT, by sandwiching resin film between two layers of dry fiber, providing air evacuation pathways, and adding slight tack to one surface for ease of layup [15,21]. This method of partial impregnation was likely inspired by early work at the Naval Surface Warfare Center, in which single-sided glass-fiber prepregs (from Newport Adhesives), with resin film applied on only one side of the reinforcement, were shown to produce low-porosity parts under vacuum pressure only, owing to air pathways provided by dry fibers [22]. In contrast to the Gurit work, Ridgard [5] described air and volatile evacuation through dry fiber “escape paths” in the MTM45-1 prepreg family (from Advanced Composites Group, now UMECO), which is partially impregnated by resin films on each side, leaving a dry fiber pathway through the center of the material. Ridgard [5] further noted that these paths must remain open for a sufficient period of time during cure, requiring careful design and control of the resin rheology. Finally, he highlighted that the initial degree of impregnation of the prepreg should be considered in light of the resin viscosity, cure cycle, and laminate quality. For example, “tightly” woven fabrics with thin tow cross-sections may must be almost entirely un-impregnated before cure to ensure that dry pathways persist for a sufficiently long time during processing.

Several studies have included images of the microstructures of commercially available VBO prepregs, as shown in Figure 3. Louis et al. [23], Grunenfelder and Nutt [24] and Wysocki et al. [25] used scanning electron microscopy (SEM) to identify dry areas within the fiber tow cores of prepregs manufactured by ACG, Cytac and Hexcel, respectively. Centea and Hubert [26] inspected the same ACG prepreg using x-ray computed microtomography (or micro-CT), and confirmed that a pervasive, continuous network of micro-porous dry fiber tows exists both initially and during an

hour-long room temperature vacuum hold. The x-ray micrographs also showed that the number and

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**Figure 3:** Examples of OoA prepreg microstructures featuring dry areas: (A) SEM micrograph of an ACG MTM45-1 5HS prepreg tow. Reproduced with permission [23]. Society for the Advancement of Material and Process Engineering (SAMPE), 2010; (B) SEM micrograph of a Cytec 5320 unidirectional tape tow. Adapted from [24]. Society for the Advancement of Material and Process Engineering (SAMPE), 2011; (C) SEM micrograph of a Hexcel Hexply M21 T tow [25]; (D) X-ray microtomography of a ACG MTM45-1 5HS prepreg laminate. Reproduced with permission [26]. Elsevier, 2011

size of entrapped air bubbles in resin-rich regions decreased during the hold, and that the dry tows were progressively infiltrated once the material was heated. The authors used the same technique to image unidirectional and woven fabric prepreps based on Cytec Engineered Materials' 5320 resin, and confirmed the same behavior [17,27,28]. Finally, Fahrang and Fernlund [29] studied the evolving microstructure of the MTM45-1/5HS by interrupted vacuum cures and optical microscopy. Three types of voids were observed: tow voids composed of inter-fiber gaps within fiber bundles, interlaminar voids formed by gaps between tows and plies, and relatively small, spherical voids in resin-rich regions. The presence of fiber tow voids was associated with a reduction in resin and

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interlaminar voids. Furthermore, the saturation of the tows was associated with a decrease of in-plane permeability, indicating that dry fiber tow cores enable the majority of the in-plane permeability.

## 2.2 Resin Properties

The properties of the resin and fiber constituents influence the evolution of VBO prepreg microstructures during cure. Generally, however, fiber properties and fiber bed architectures are standardized, whereas matrix properties drive both prepreg and process development [9]. The dependence of microstructural evolution on resin properties, therefore, is critical to understand, and has been investigated by numerous authors.

The presence of dry prepreg areas may suggest a need for low-viscosity resins. However, Ridgard [5,6] explains that VBO prepreg systems are designed to remain relatively viscous in the early stages of cure to impede infiltration and allow sufficient dry areas to persist for air evacuation to occur. Because the room temperature vacuum holds used to evacuate air from VBO systems are sometimes measured in hours or days, it is critical for the resin viscosity to inhibit “cold flow”, which could prematurely seal the air evacuation pathways [16]. However, the overall viscosity profile must also permit sufficient flow at cure temperature to fully impregnate the prepreg, lest pervasive dry areas remain in the final part [16]. Furthermore, Boyd and Maskell [7] argue that to inhibit bubble formation and growth at low consolidation pressures, both the viscous and elastic characteristics of the prepreg must be tuned to the specific processing parameters encountered during cure, and ultimately ensure that a majority of the applied pressure is transferred to the resin. Altogether, the rheological evolution of VBO resins must balance the reduction of both voids caused by entrapped gases and voids caused by insufficient flow.

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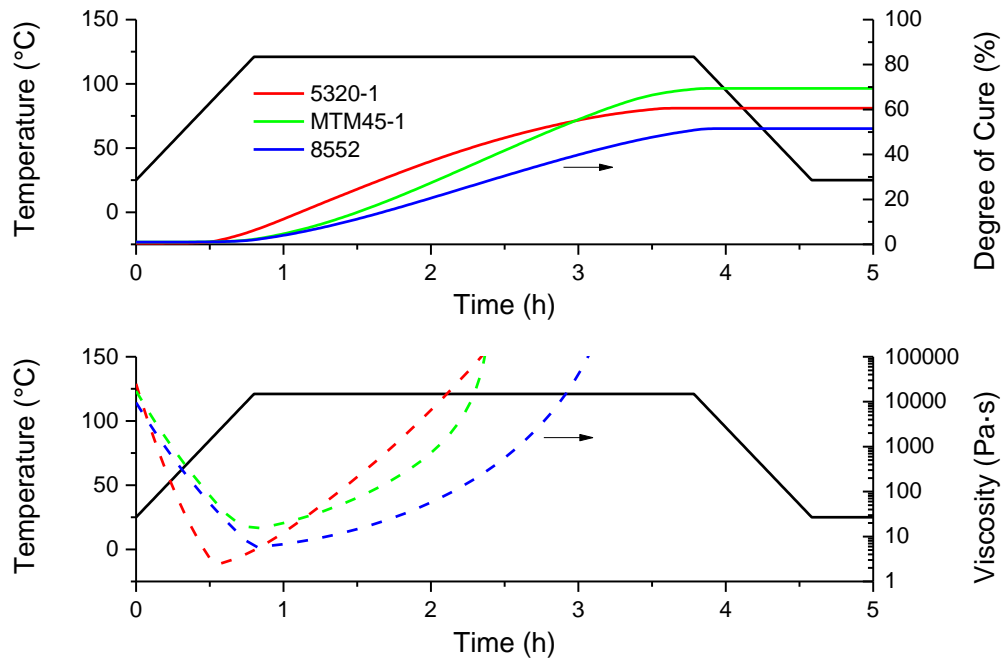
Multiple authors have investigated aspects of the VBO prepreg microstructural evolution. For example, Lee et al. [30] developed reaction rate and viscosity models for a resin matrix (Cytec's Cycom 5320-1) using experimental data from differential scanning calorimetry (DSC) and rheometric dynamic analysis (RDA), along with two predictive approaches: look-up tables and neural networks. Both reaction rate models showed acceptable performance, with the neural net approach capturing the second half of the reaction (and thus the shift to diffusion-controlled cure) more accurately. Likewise, Kratz et al. [31] characterized the cure kinetics, viscosity and glass transition temperature for two commercial VBO prepregs (ACG's MTM45-1 and Cytec's Cycom 5320). DSC, RDA, and dynamic mechanical analysis (DMA) experiments were carried out in various process conditions, and the resulting data was used to develop semi-empirical analytical models that effectively predicted these properties for any time-temperature cycle. Soltani et al. [32] developed a similar mode for Cytec's Cycom 5320 system based on DSC measurements and an existing cure kinetics model. The developed equations predicted the degree of cure evolution at isothermal conditions accurately, but had to be adapted individual staged cure cycles for close agreement. Finally, Kim et al. [33] characterized the thermochemical properties of another resin system (Cytec's Cycom 5320-1) in a similar manner to Kratz et al. [31]. These studies highlight commonalities between VBO prepreg resin systems. These resins exhibited traditional cure kinetics behavior, with an initial increase in the rate of cure from progressive cross-linking, followed by an eventual decrease resulting from a shift to diffusion-based mechanisms. Intermediate temperatures (80°C to 120°C) gelled and vitrified the resins, but achieving complete polymerization and a maximal degree of cure required a higher-temperature post-cure. Rheology results for the aforementioned systems showed that minimum viscosities range from approximately 1 Pa·s to 10





Pa·s for dynamic ramps and 1 Pa·s to 100 Pa·s for isothermal conditions, and that gelation takes place during the intermediate dwell in all manufacturer-prescribed cure cycles.

Kratz et al. [31] also compared two VBO resins (MTM45-1 and 5320) to a common autoclave system (Hexcel's HexPly 8552). As expected, both VBO resins were more reactive than the autoclave resin in the 80°C to 140°C range, as the later was intended for 177°C cure. Interestingly, however, because of this higher reactivity, and generally higher viscosities, both VBO systems offered only 60 min to 120 min of "flow time" (defined by the authors as time spent at a viscosity below 100 Pa·s) rather than the 500 min offered by the autoclave resin, suggesting that past the onset of cure, the period available for full prepreg impregnation is limited. A comparison of three resin systems, two VBO (Cycom 5320-1, ACG MTM45-1) and one autoclave (HexPly 8552), based on published models [31,33,34], is shown in Figure 4 and reveals that the autoclave prepreg is less reactive and less viscous for most of the 121°C cure dwell time. Note that none of these resin systems is fully cured after a three-hour dwell at 121°C. The two VBO systems are designed to undergo a freestanding post-cure at 177°C., while the autoclave resin is intended for 177°C cure (not shown).



**Figure 4:** Comparison of the evolution of the degree of cure ( $\alpha$ ) and viscosity ( $\mu$ ) for the Cytec Cycom 5320-1, ACG MTM45-1 and Hexcel HexPly 8552 resins.

Most published work on VBO prepreps has utilized commercially available product forms. In one example, however, academic researchers developed a novel curing agent, which was combined with commercial epoxies to produce a low-temperature-curing resin suitable for vacuum bag processing [35]. The resin system was incorporated into prepreps and cured to make composite laminates. The main challenge encountered was the impregnation of the fiber preform by the resin system, which was improved through the use of ultrasonic agitation [35].

Owing to reduced processing pressures, VBO resins and prepreps are susceptible to environmental effects, as shown by Grunenfelder and Nutt [36–38]. The authors first considered the effect of absorbed moisture, and showed that exposure to high relative humidity can reduce the gel





time of the resin [36]. In this work, a commercial VBO resin (ACG's MTM44-1) was conditioned for 24 h at 35°C, and various humidity levels. An increase in ambient humidity from 50% to 90% corresponded to a decrease in gel time from 160 min to 130 min. The authors also considered the effect of out-time on the cure kinetics and tack of VBO prepregs (Cyttec's Cycom 5320) [37]. Both properties were strongly impacted. Modulated differential scanning calorimetry (MDSC) data indicated that out-time decreases the heat of reaction and increases both the uncured and cured (ultimate) glass transition temperature ( $T_g$ ) in a quasi-linear manner. Energy of separation tests indicated that the tack remains relatively unchanged over the first 14 days, but then quickly decreases to zero.

In a subsequent publication, Grunenfelder and Nutt [38] also applied the MDSC out-time monitoring technique to two other systems, Cyttec's Cycom 5320-1 and ACG's MTM44-1, and showed that the uncured  $T_g$  is a reliable metric for tracking prepreg out-time. Over 56 days, the uncured  $T_g$  of the prepreg increased from about 0°C to approximately 20°C to 40°C, and the estimated degree of cure increased from 0 to between 0.10 and 0.30, depending on the material. Similar results were obtained by Kim et al. [33], who studied the cure kinetics and viscosity of the Cycom 5320-1 resin from 0 days to 49 days of out-time, in weekly increments, and confirmed significant changes in both the cure rate and the viscosity. In complementary work, Jones et al. demonstrated the utility of photoacoustic spectroscopy to monitor the ambient aging of an OoA material system (Toray T700SC-12K-50C/#2510 plain weave fabric prepreg) [39]. Similarly, Kim et al. [33] developed correlations between the degree of cure and viscosity of the Cycom 5320-1 resin and dielectric signals obtained from prepreg samples, and demonstrated that dielectric analysis (DEA) may be used for in-situ cure monitoring.



## 2.3 Fiber Bed Properties

Relative to the literature on resin properties, published studies on the fiber beds used in VBO prepregs are scarce. However, as explained by Bond et al. [9], resin development drives prepreg development, and VBO prepregs feature a wide but standard variety of fiber beds.

The fiber volume fraction of a cured laminate is governed by the compaction curve of the fiber bed and the load sharing between the fiber bed and the resin at gelation. During VBO processing, the compaction pressure applied on the laminate is, in the ideal case, constant and equal to the ambient pressure. Conversely, during autoclave processing, the applied pressure is several times higher and may theoretically be adjusted to modify the ultimate fiber volume fraction of the part. For these reasons, it is not surprising that slight differences have been reported in the thickness and fiber volume fraction of parts cured with the same material in and out of an autoclave [40]. Fiber volume fractions on the order of 60%, however, are readily achievable with VBO prepregs, a benefit over many infusion-based OOA methods [40,41].

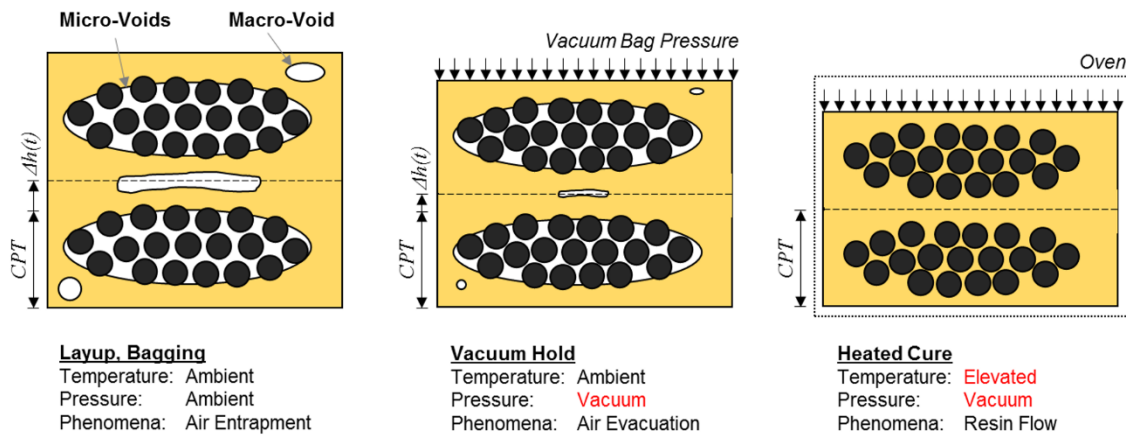
Ridgard [4] does caution that some product form limitations may exist due to the finite set of viable combinations of reinforcement fiber volume fraction, weight and size, and resin rheology and cure cycle. For example, a fabric with high areal weight and a low degree of impregnation may require a lower resin viscosity profile to achieve full wet-out prior to resin gelation, and this profile may not be achievable for all resin chemistries and temperature cycles. Thus, while all fibers and architectures are potentially relevant to VBO prepregs, their selection in a given prepreg, part and process must be considered in light of other manufacturing constraints.



### 3. Processing

#### 3.1 Consolidation

Consolidation studies are generally concerned with the behavior of the three composite phases – fibers, resin, and voids – during processing, and with their final state at the end of cure. For VBO prepregs, these phenomena include air evacuation, resin flow and fiber bed compaction, and void formation. The OoA prepreg consolidation process is shown schematically in Figure 5 for adjacent unit cells consisting of elliptical fiber tows and surrounding resin. Initially, after layup, a laminate consists of an unconsolidated stack of partially impregnated plies. Each ply consists of aligned fibers (or fiber tows), surrounding resin rich regions, micro-voids within the dry tow cores (between individual fibers), and macro-voids within the resin-rich regions located around the tows or between plies. Once vacuum is applied, air is evacuated through the dry tow cores, the macro-void content decreases, and the fiber bed is compacted. However, the tows remain partially dry. Once the temperature is raised, the resin gradually infiltrates the dry tow areas to produce (ideally) a void-free microstructure (for reinforcements with crimp, some flow along the fiber direction may also occur). The thickness of each ply reduces by an amount  $\Delta h(t)$  to a uniform final cured ply thickness (CPT), in the absence of voids.



*Figure 5: Schematic of the OoA prepreg consolidation process, showing unit cells consisting of aligned fibers (or fiber tows), surrounding resin, micro-voids within the tow cores and macro-voids within resin-rich regions. CPT indicates cured ply thickness.*

The nature and rate of these phenomena, as well as the effects of key material properties and process parameters, have been the topic of numerous publications. Generally, the formation and mitigation of voids is the primary concern. For clarity, voids are classified as *flow-induced*, resulting from the inability of the resin to fully impregnate the dry areas before gelation, and *gas-induced*, formed by the presence of entrapped air, vaporized moisture or cure-generated volatiles.

### 3.1.1 Air Evacuation

Arafath et al. [42] described gas transport in prepregs analytically as the unidirectional flow of a compressible fluid through a permeable porous medium, and used the resulting model to analyze the flow through two Toray unidirectional and woven fabric autoclave prepregs. The equations, based on Darcy's Law and the ideal gas law, non-dimensionalized, predict the time required to evacuate a given mass fraction of gas. These equations also clarify the physical laws of evacuation, showing that under the invoked assumptions, evacuation time increases linearly with air viscosity

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and quadratically with part length, and decreases linearly with fiber bed permeability. These relations indicate that if, for example, the distance the air must travel leaving the prepreg doubles (due to a longer laminate or impermeable edges), the required evacuation time quadruples.

Several authors have directly characterized the air permeability of VBO prepregs. For example, Louis et al. [23] imposed steady-state flow and determined the in-plane and through-thickness permeabilities of a VBO prepreg (MTM45-1/5HS) under room temperature vacuum compaction by measuring the mass flow rate and assuming Darcy flow. The results showed that in-plane permeability is up to four orders of magnitude greater than transverse permeability, at  $10^{-14} \text{ m}^2$  versus  $10^{-18} \text{ m}^2$ , respectively. Furthermore, the data indicated that flow in the transverse direction shows non-Darcian behavior, with the permeability decreasing to nearly zero as plies are added. The authors hypothesize that the deviation from Darcy's Law is caused by non-uniform through-thickness gas flow, which occurs predominantly through a few resin-starved areas. They argue that when multiple plies are used, these "holes" are increasingly misaligned, creating a discontinuous pore network and decreasing the effective permeability. Finally, the authors proposed that ply terminations restrict gas flow to the equivalent of the thinnest region of the laminate. As previously noted, Fahrang et al. [29] measured the in-plane permeability of the same material during a single process cycle. Their results agreed with Louis et al., showing an order of magnitude of  $10^{-14} \text{ m}^2$ , and further showed that in-plane gas transfer decreases to zero as the degree of impregnation of the tows progresses. Finally, Grunenfelder and Nutt [24] used a falling-pressure method to estimate the permeability of unidirectional tapes (Cycom 5320). The unidirectional material showed greater in-plane permeability along the fiber direction, where pathways for air removal are direct, with permeability  $1.5 \times$  less in the  $90^\circ$  direction (perpendicular to the fibers), where air flow is more

tortuous. Sixteen-ply unidirectional panels 560 mm in length were produced, breathing from one

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edge only, with non-permeable dams around the remaining edges. A greater void content was observed in the 90° panels, accompanied by an increase in thickness (reduction in compaction) as a function of distance from the breathing edge, indicating an interplay between fiber bed compaction, permeability and air evacuation.

In a more complex context, several authors have characterized air evacuation in honeycomb sandwich structures, within which prepreg laminate facesheets must accommodate the transverse flow of the air entrapped within the honeycomb cells. Literature specific to the processing of honeycomb structures will be addressed in detail in section 3.2.2. However, the following results are generally relevant to prepreg air evacuation.

Sequiera-Tavares et al. [43] used a custom-designed apparatus consisting of a metal mold with a machined pocket of known volume to determine the through-thickness permeability of a unidirectional tape prepreg (ACG's VTM264) through the falling pressure method. Tests were performed at both room temperature and cure conditions. The room-temperature permeability was reported as  $5 \times 10^{-18} \text{ m}^2$ , in agreement with Louis et al [23]. During cure, the permeability decreased because of progressive prepreg impregnation during an initial temperature ramp and dwell, before increasing and stabilizing during a second ramp as a result of decreasing viscosity and, ultimately, gelation. The study indicated that the through-thickness air evacuation capacity evolves during processing based on both resin and prepreg factors.

Kratz and Hubert [44] and Kratz et al. [45] used a similar experimental set-up to determine in-plane and out-of-plane permeabilities for prepregs consisting of a VBO resin (Cycom 5320) and plain weave (PW) and 5HS fabric architectures. The areal weight and “tightness” of the fabric architecture had a major impact on transverse permeability. The relatively lightweight PW (196

$\text{g/m}^2$ ) featured regular gaps between tows and exhibited consistent permeability as a function of

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laminate thickness. Conversely, the heavier 5HS ( $375 \text{ g/m}^2$ ) had practically no open macro-spaces between tows, and exhibited erratic permeability as well as delays in the onset of flow of up to 24 h in thick laminates.

Two major points emerge from this literature. The first is that average in-plane permeabilities are relatively low in comparison to other porous materials, and the room temperature vacuum hold times required to evacuate a majority of entrapped air are relatively long in the context of prepreg cure. For example, according to the model by Arafath et al. [42], for an evacuation length of 1 m and an in-plane permeability of  $1 \times 10^{-14} \text{ m}^2$ , removing 90% of the air takes approximately 30 h. The second is that through-thickness evacuation, which may ostensibly be more efficient because of short flow lengths, is similarly challenging because the through-thickness permeability is more than three orders of magnitude less than in-plane, and certain product forms can exhibit erratic and non-Darcian transverse flow with increased laminate thickness. Average room-temperature prepreg permeabilities are shown in Table 2 for various VBO material systems.



**Table 2:** Average in-plane and through-thickness room temperature permeabilities reported in the literature. The data, while based on different prepregs, shows a three-order-of-magnitude difference between in-plane and transverse values.

DIRECTION	MANUFACTURER	RESIN	FIBER BED	PERMEABILITY [m <sup>2</sup> ]	REFERENCE
In-Plane	ACG	MTM45-1	5HS	$3.3 \times 10^{-14}$	[23,29]
	Cytec	5320	5HS	$8.5 \times 10^{-15}$	[44]
	Cytec	5320	PW	$7.3 \times 10^{-15}$	[44]
	Average			$1.6 \times 10^{-14}$	
Transverse	ACG (now Cytec)	MTM45-1	5HS	$1.0 \times 10^{-18}$	[23]
	Cytec	5320	5HS	$1.6 \times 10^{-17}$	[44]
	Cytec	5320	PW	$6.4 \times 10^{-17}$	[44]
	ACG	VTM264	UD	$5.0 \times 10^{-18}$	[43]
	Average			$2.5 \times 10^{-17}$	

### 3.1.2 Flow and Compaction

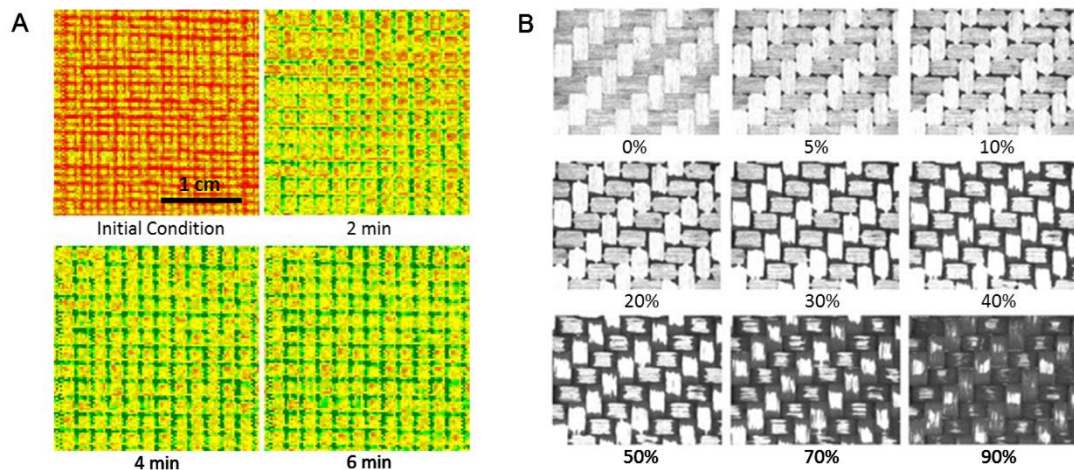
Fiber bed compaction, resin infiltration, and the consequent evolution of the dry and resin-rich areas have been analyzed from a wide variety of perspectives. For example, Thomas et al. [46] and Thomas and Nutt [47] used ultrasound to monitor vacuum-driven through-thickness resin film infiltration into a carbon fabric, first demonstrating the method under isothermal conditions and then extending it to multiple temperatures. Their results are relevant to both the prepregging and cure of VBO prepregs, and indicate that flow occurs over dual length- and time scales. First, resin is driven into the larger inter-tow macro-spaces, leading to a microstructure of dry tow cores and surrounding resin-rich regions. Then, resin flows into the smaller intra-tow micro-pores, completing impregnation (Figure 6A). The significant influence of temperature (or resin viscosity) on flow times is clearly demonstrated: for the studied resin (Cycom 5215), infiltrations at 50°C and 80°C correspond to impregnation times of 6 min and 48 min, respectively.

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Cender et al. [48] carried out a similar study by curing one-sided prepregs made of dry fabric and resin film (Gurit Single Sprint ST94-RC200T and ST940RC303T) over a transparent mould in various pressure and temperature conditions. The pattern of dry and infiltrated regions at the tool-part interface indicated the presence of dual scale flow, with initial infiltration of inter-tow areas (as during prepregging) and subsequent infusion into the tow cores (Figure 6B). For the studied fabric reinforcement, intra-tow flow occurred in both the axial and transverse tow directions, and a slow-down in the rate of infiltration at high impregnation levels indicated that significant load-sharing between resin and fibers may have taken place. The authors proposed a model describing inter- and intra-tow flow prior the onset of load-sharing as one-dimensional infiltration through two successive porous media, which showed agreement with experimental data.



**Figure 6:** (A) Ultrasound scans showing impregnation as a function of time in a VBO system at 80° C. Reproduced with permission [47]. Springer, 2009; (B) Images showing dual-scale flow in VBO prepreg as percent impregnation increases, with initial flow occurring at tow overlaps, followed by impregnation of tows. Reproduced with permission [48]. Elsevier, 2013.



In aforementioned studies, Fahrang et al. [29] and Centea and Hubert [26,27] partially processed commercial OoA prepregs (MTM45-1 and 5320) resins to different stages of representative cure cycles and used optical microscopy and micro-CT, respectively, to investigate the impregnation state. Both determined that the initial microstructure of the prepregs consists of dry tow cores surrounded by resin-rich regions, and identified tow impregnation at elevated temperatures as the key flow phenomenon during cure.

A complex modeling framework applicable to partially impregnated prepregs was developed in successive studies by Larsson et al. [49], Wysocki et al. [25] and Rouhi et al. [50,51]. The method uses a two-phase continuum, porous media theory formulation to capture fiber bed deformation as well as coupled resin flow at both the micro-scale (inter-tow) and macro-scale (intra-tow and ply) levels. This formulation can be implemented in finite element analysis for detailed studies. Several applications are used to demonstrate the capacities of the approach, including (a) volumetric relaxation, creep, and no-bleed compression [49], (b) the consolidation of a hat stringer [25], and (c) drained and undrained compression tests [51]. The models are shown to capture the evolution of constituent fractions, saturations, and reaction forces.

A simplified analytical model for tow impregnation under non-isothermal conditions was developed by Centea and Hubert [27] based on Darcy flow within a rigid circular porous medium. Model parameters were determined for three VBO prepregs (MTM45-1, 5320/8HS and 5320/PW), and validated for benchmark cure cycles using micro-CT. The models were then used in a parametric study to evaluate the effect of various material and process factors on tow impregnation rate. The results indicated that tow impregnation depends strongly on the resin viscosity profile, and therefore on the cure cycle ramp rate and dwell temperature and the resin initial degree of cure (or out-time), as well as on the fiber volume fraction and transverse permeability of the tows.

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Gangloff et al. [52] modeled the compaction and saturation of partially impregnated prepreg tape during automated deposition or vacuum consolidation. The model utilized one-dimensional porous media flow theory and material constitutive equations to predict the evolution of the degree of resin saturation in the tape and the prepreg thickness under constant or transient compaction. With this approach, the remaining degree of dry fiber regions available for gas evacuation during subsequent part cure could be estimated. The effect of various deposition and compaction factors was studied. The results indicated that, generally, the constant pressure applied by vacuum consolidation causes increased resin saturation and thickness reduction, whereas the comparatively transient compaction during automated deposition can allow material in the remaining dry regions to “spring back” after compression, leading to lower final deformation. In both cases, increased pressure was shown to increase resin saturation levels and decrease dry fiber areas. The above literature shows that VBO prepreg consolidation is a dual-scale phenomenon, consisting of macro-scale flow between plies and around tows, and micro-scale flow within the tows. For prepregs with fabric reinforcements, flow at these two length scales takes place at different times and rates. Within each ply, the macro-pores are generally saturated with resin during preprepping, particularly if, as with most studied materials, each prepreg ply features resin on both sides. Therefore, within a laminate, the inter-tow spaces are resin-rich, and the key remaining flow phenomenon is flow into the tows. For a unidirectional prepreg, separate macro- and micro-scale flow may also occur if the reinforcement consists of discrete adjacent tows (such as those within unidirectional non-crimp fabric) rather than a single volume of aligned fibers (such as in traditional unidirectional tape). In both cases, the literature suggests that the eventual tow impregnation can be influenced by numerous material and process parameters. Several studies have considered these relationships and their effect on defect formation.

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### 3.1.3 Void Formation

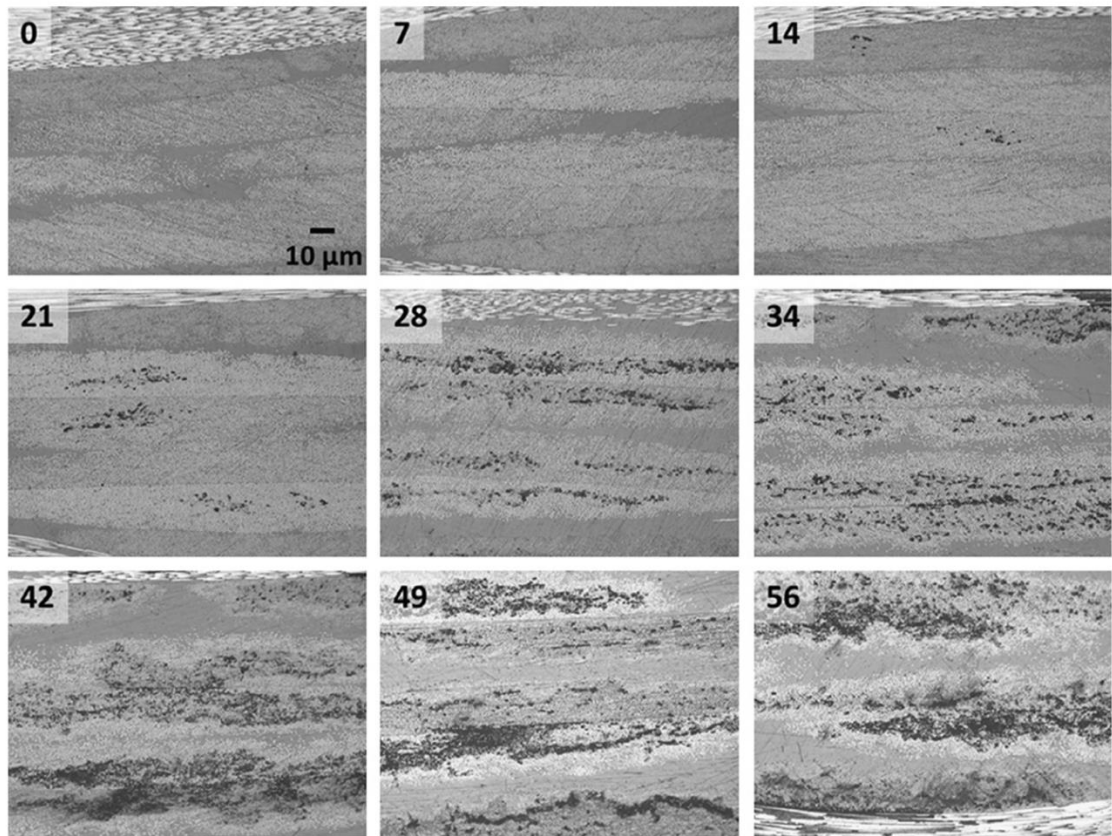
Multiple sources of void formation exist in the fabrication of composite parts, including entrapped air, insufficient resin flow, and evolved gasses. Autoclave pressures alleviate many of these issues, whereas void formation under reduced VBO consolidation requires a more thorough understanding.

#### 3.1.3.1 Flow-Induced Voids

The parametric study carried out by Centea and Hubert [27] indicated that cure cycle temperature and resin initial degree of cure can have a significant effect on the time required to fully impregnate fiber tows. In particular, combinations of fiber-dense tows, slow heat-up rates, low dwell temperatures, and high initial degrees of cure (induced, for example, by out-time) can lead to incomplete impregnation before gelation, and pervasive micro-porosity. However, for the same fiber and resin properties, high dwell temperatures and fast ramp rates can mitigate this danger by decreasing the minimum viscosity.

Grunenfelder et al. [17] directly compared the same model's predictions with measured data from laminates made of prepreg (5320/5HS) cured under a single temperature cycle at zero to eight weeks of out-time. Both model and experiments showed that tow porosity begins to form once the resin out-life is exceeded (in this case, after 21 days, as shown in Figure 7). Following the onset of tow voids, porosity increases in size and prevalence until the resin's glass transition temperature exceeds the ambient temperature, and cross-linking ceases due to reduced molecular mobility. The model under-predicted the size of the unsaturated tow area by approximately 33%, either due to an inability to accurately predict when flow ceases or because of unaccounted porosity sources (such as entrapped air or vaporized moisture within the tows). However, the predicted trends confirmed

the hypothesis that the underlying cause of out-time-induced porosity is caused by flow phenomena. Please cite this article as: Centea T, Grunenfelder LK, Nutt SR. **A review of out-of-autoclave prepreps - material properties, process phenomena and manufacturing considerations.** *Compos Part A Appl Sci Manuf* [Internet]. 2014; 70:132–154. Available from: <http://www.sciencedirect.com/science/article/pii/S1359835X14003108>



*Figure 7: Porosity as a function of out-time in a VBO prepreg. Following the manufacturer's stated out-life (21-days) intra-tow porosity is observed as a result of insufficient resin flow. Reproduced with permission [17]. Elsevier, 2013.*

The influence of out-time on porosity in parts produced from two VBO resin systems was also examined by Lucas et al. [11]. The Cycom 5320 system was exposed to out-times of up to 20 days, showing increasing porosity with out-time. To extend out-time and combat associated issues, Cytec developed a modified resin system, denoted 5320-1. This material was formulated for longer out-life, requiring an extended cure time. Laminates made with this resin system showed acceptable void content of less than 1 % up to out-times of 33 days. Contrary to the study by Centea et al. [27],

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greater void contents were observed with faster heating rates, suggesting that slow ramp rates may be beneficial to VBO cure, though the underlying mechanisms of the observed porosity are unknown.

Subsequently, Centea et al. [53–55] also carried out experiments on the relationships between cure cycle, out-time, and fiber bed architecture [53,54], as well as ply orientation and stacking sequence [55]. The results confirmed that rapid, high-temperature cure cycles induced faster consolidation (relative to the start of the dwell period) and, in cases of long out-time, mitigated or even eliminated flow-induced micro-porosity. The results also confirmed that out-time reduces prepreg tack and can cause ply warpage, but that such changes can also improve the air evacuation capacity of the laminate and reduce gas-induced macro-porosity. Finally, the uneven surface morphology of fabric plies applied uneven pressure on adjacent layers, but ply orientation and stacking sequence showed no effect on void distribution and porosity as long as the prepreg out-time was not exceeded.

Overall, these studies emphasize that under VBO consolidation, partial impregnation renders VBO prepregs susceptible to significant and potentially catastrophic flow-induced micro-voids if material and thermal conditions combine to shift the resin viscosity too far from the designed range. These conditions include excessive out-time (or intermediate out-times and low-temperature cure), and form an intrinsic process limit for such materials. Flow-induced voids are purely a function of fiber bed and resin properties, and can occur regardless of how well gas-induced void sources, such as those outlined below, are controlled.

### *3.1.3.2 Gas-Induced Voids*

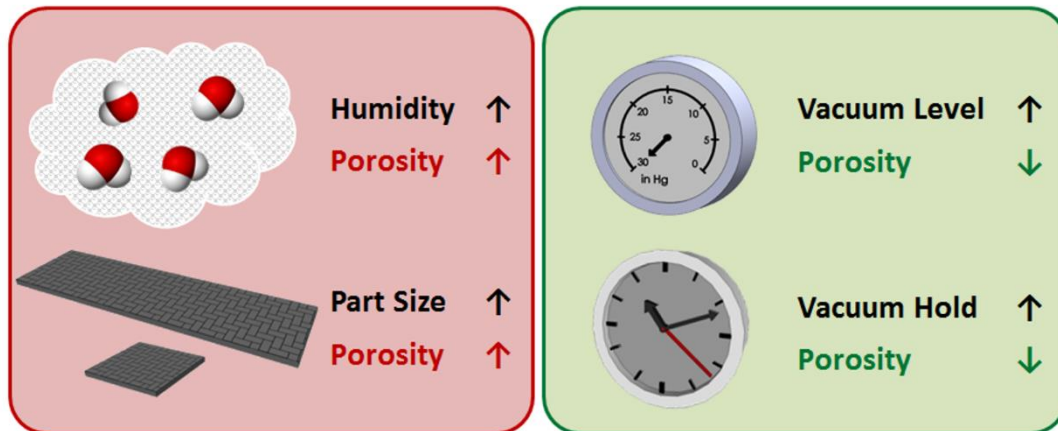
Grunenfelder et al. [36] exposed prepreg to elevated ambient relative humidity conditions, and measured a quasi-linear increase in moisture weight percentage within the resin and an

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exponential increase in void content for VBO-cured laminates. Conversely, void contents in autoclave-cured laminates remained low for all humidity levels. A diffusion-based void growth model, based on a framework proposed by Kardos et al. [56], was developed and used to explain these trends by showing that the gas pressure within moisture-induced voids can exceed the maximum resin pressure attainable with VBO cure at cure temperatures of  $\sim 120^{\circ}\text{C}$ , while autoclave-induced resin pressures remain above void pressures, suppressing moisture-based void formation.

Fernlund and colleagues [57,58] carried out experimental studies on the influence of ambient relative humidity, vacuum level, room temperature hold time, and part length on porosity. Increased relative humidity and reduced vacuum levels both increased void contents individually, and exacerbated each other when combined. Both effects were ascribed to a lower ratio of resin pressure to void pressure. For the same vacuum hold times, a longer flow distance for air evacuation increased porosity, compounded the effect of relative humidity exposure, and created a porosity gradient along the evacuation direction. All effects were attributed to larger and more numerous entrapped air sites and more difficult air evacuation. Interestingly, for a long part, porosity was fully eliminated in the case of exposure to 0% relative humidity and a long (24 h) vacuum hold, confirming that for the studied prepreg, moisture and entrapped air were the dominant causes of gas-induced voids within OoA prepreps, and negating the possibility of cure-induced volatiles. Qualitative representations of the influence of various process parameters on void content are presented in Figure 8.



*Figure 8: Influence of various process parameters on porosity in VBO composites. Adapted from [58].*

Centea and Hubert [28] studied the effect of ambient pressure, vacuum quality, restricted air evacuation, and fiber bed architecture on porosity. Reduced ambient pressure and vacuum quality produced comparable and quasi-linear increases in voids within resin-rich regions because of a lower ratio of resin pressure to void pressure. However, reduced vacuum also led to micro-porosity within the tows by impeding resin infiltration, a result consistent with prior studies [21]. An extreme case of restricted air evacuation was achieved by sealing the laminate edges and produced catastrophic porosity, indicating that entrapped air must be evacuated in-plane. Intriguingly, in all cases but the latter, unidirectional tape laminates contained considerably less porosity and smaller individual voids than fabric-based laminates, indicating that fiber beds that entrap more air, such as fabrics, are significantly more sensitive to deviations from ideal process conditions.

Overall, existing research confirms that, in the absence of an elevated pressure safeguard, VBO prepreps are vulnerable to any process deviation that reduces their void-suppression capacity, either through decreased resin pressure (from reduced ambient pressure) or, more likely, through

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increased void pressure caused by inadequate vacuum, insufficient air evacuation, or moisture vaporization. This sensitivity, in turn, motivates the need for strict material storage, handling and cure protocols.

### 3.2 Mechanical Properties

With proper processing techniques, VBO cured laminates have been produced that show property equivalence to panels produced in an autoclave. Many early generation VBO resins were adapted from autoclave curing systems. One such resin (TenCate's TC 350-1) was processed using both VBO and autoclave methods, and mechanical properties were examined [59]. Tensile, compressive, and flexural properties were equivalent in both sets of panels. A unidirectional VBO prepreg developed by another manufacturer was also cured both using vacuum pressure only and in an autoclave [40]. In this case, oven-cured panels showed greater thickness and void content than autoclave cured samples, accompanied by a decrease in matrix-driven mechanical properties.

Mechanical property equivalence to traditional autoclave materials has also been shown for novel VBO systems. First-generation Cytec VBO prepregs (5215 and 754), for example, were shown to have property equivalence with traditional autoclave systems in terms of glass transition temperature as well as tensile, compression, short-beam shear and compression after impact properties for both woven fabric preforms and unidirectional tapes [8]. Property equivalence has also been demonstrated for later generation prepregs (5320 and 5320-1) [11].

NASA performed a detailed study examining the mechanical properties of autoclave (IM7/8552-1 and IM7/977-3) and out-of-autoclave (IM7/MTM45-1 and T40-800b/5320) unidirectional prepregs, taking into account material out-time as well as hand lay-up vs. fiber-placement techniques

[41]. Laminate quality was assessed in terms of void content and mechanical properties, focusing on Please cite this article as: Centea T, Grunenfelder LK, Nutt SR. **A review of out-of-autoclave prepregs - material properties, process phenomena and manufacturing considerations.** *Compos Part A Appl Sci Manuf* [Internet]. 2014; 70:132–154. Available from: <http://www.sciencedirect.com/science/article/pii/S1359835X14003108>



resin-dominated properties of short-beam-shear strength, compression, and open-hole compression. VBO prepregs were shown to be more sensitive to out-time than autoclave systems, displaying decreases in tack and drape accompanied by high void contents and reduced mechanical properties in cured laminates. Preliminary results indicated that panels cured using fresh VBO prepreg, however, had properties equivalent to autoclave processed parts in the tests performed.

The effect of certain process VBO process parameters on mechanical properties has also been studied. Vo et al. [60] investigated the effect of post-cure temperature variations on the resin-dominated compressive properties of a VBO prepreg (Cytec's 5320/8HS). Panels were manufacturing using a non-standard cure cycle consisting of a two-hour cure at 93°C and a post-cure at temperature ranging from 99°C to 143°C, while DSC, DMA, combined loading compression (CLC) testing and fractography were used to analyze processing and performance. The results showed a strong correlation between degree of cure, CLC strength and glass transition temperature, with all three properties increasing with higher post-cure temperature, as well as a shift in failure mode from resin-dominated to resin-and-fiber dominated, indicating improved resin properties and fiber-resin bonding. Walker et al. [60] complemented this study by analyzing the correlation between cure state and room temperature short-beam shear strength. Panels were similarly manufactured and tested, while the viscoelastic evolution of the prepreg's properties was measured using an encapsulated shear rheometer. In addition, hygrothermal conditioning was used to investigate the effect of hot/wet environments. As in the prior study, the mechanical strength, dry glass transition temperature and average storage modulus increased with higher post-cure temperature, with the short-beam shear and glass transition data showing the strongest inter-relation.

Gernaat et al. [61] studied the relationship between several viscoelastic properties and certain resin-dominated mechanical properties for a first-generation VBO prepreg (Cycom 5215 with a plain

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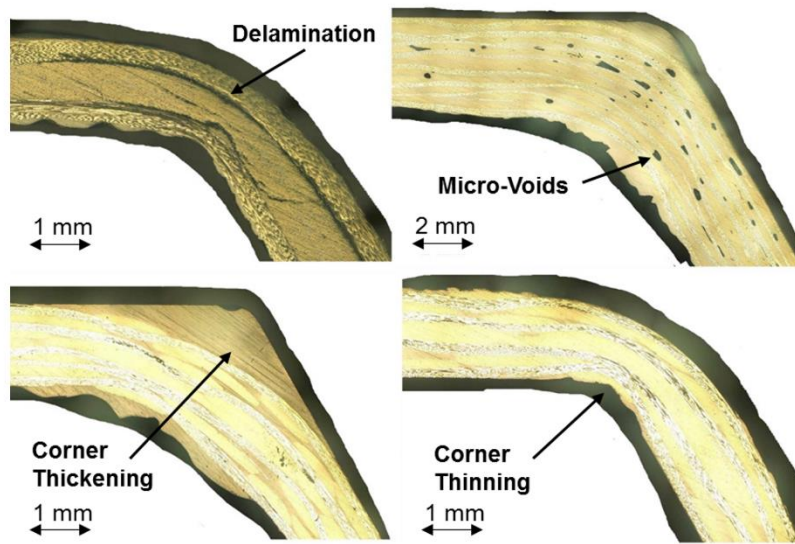


weave fabric). Viscoelastic properties (storage modulus, glass transition temperature) were measured during cure by shear rheometry. Five panels were manufactured using VBO oven cure, and tested using CLC to determine compressive modulus, strength and Poisson's ratio. The results show a correlation between the value of the storage modulus at the end of cure and the compressive-mechanical properties, suggesting that for this VBO material, the storage modulus may be a reliable means of non-destructively evaluating the mechanical properties of a part cured using a non-standard temperature cycle.

These studies provided initial indications of the ability to produce autoclave quality parts using VBO methods. These works, however, and the majority of the investigations discussed thus far, relate to the manufacture of small, flat panels. The manufacture of large composite parts and parts with complex geometries present additional challenges.

### 3.3 Complex Geometries

Composite structures often contain curvatures and other geometric features that complicate the layup process and introduce additional physical phenomena during consolidation. The flow and compaction phenomena occurring near such features are more complex than those that occur in flat laminates, and may lead to defects that are not observed in flat laminates, such as those shown in Figure 9. For ease of discussion, work pertaining to these topics has been divided into three categories – (1) parametric studies, which evaluate the influence of one (or a few) complex features on physical phenomena and part quality, (2) studies pertaining to the manufacture of honeycomb sandwich panels, and (3) demonstrator studies, which report on the fabrication of realistic structures with multiple challenging features.



**Figure 9:** Examples of defects observed in OOA/VBO prepreg laminates with complex geometries ( $60^\circ$  angles with sharp mold corner radii) [62].

### 3.3.1 Parametric Studies

Soltani et al. studied the effect of fabric architecture and layup on out-of-plane distortion in VBO prepregs. In a first study [63], four commercially-available were used to produce single ply and 16-ply asymmetric balanced flat laminates by VBO oven cure, which were then accurately measured using a laser probe coordinate measurement machine (CMM). The reinforcement architectures consisted of two unidirectional tapes, an eight harness satin woven fabric and a biaxial non-crimp fabric. The single-ply non-crimp fabric laminate exhibited the most distortion due to its inherent reinforcement anisotropy, whereas the single-ply unidirectional laminate was the flattest. In contrast, the 16-ply unidirectional laminate showed the most distortion among the thicker parts, whereas the woven fabric laminate showed the least. In a subsequent study [64], the authors used a design of experiments (DOE) approach to estimate the importance of non-woven fabric type, layup symmetry

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and layup angle count on distortion. Eight panels were manufactured and characterized using CMM. The analysis identified layup symmetry as the dominant factor, indicated that the fabric time and layup angle were five to six times less influential, and highlighted some two-way interactions between the three factors. Altogether, symmetric layups, non-crimp fabric prepregs and high angle counts reduced distortion.

Brillant and Hubert [65] investigated the processing of L-shaped laminates, with particular emphasis on quality at the 90° angle. Parts were manufactured on both concave and convex tools, with and without pressure-intensifying silicone strips of various sizes. The concave tool led to noticeable resin accumulation and relatively high porosity at the corner, while the convex tool produced better quality and more uniform parts. However, a pressure-intensifying strip placed at the corner reduced such defects on convex tools. The influence of this pressure enhancement was explained through a simple finite-element simulation showing that the strip is able to “transfer” loads from the flanges to the corner, rendering the compaction stress distribution within the part more uniform.

The manufacture of 90° angles was also examined by Luner and Bond [66], using a design of experiments approach. Three woven fabrics - a five-harness satin, eight-harness satin, and crowfoot satin weave - were examined using male tooling. A unidirectional tape containing the same resin system was also investigated using both male and female tooling, with varying bend radii. VBO cured samples were shown to have less “spring in,” or reduction in angle, after cure than autoclave processed parts, indicating lower residual stresses. Reinforcement type, tool radius, and cure temperature were shown to influence part quality. Specifically, increasing tool radius resulted in increased thickness and load-bearing capability, and lower cure temperatures led to reduced “spring



in.” Radius type (male vs. female) produced significant differences in unidirectional samples, with female tooling resulting in fiber bridging and lack of compaction pressure during cure.

Cauberghs and Hubert [67] studied the effect of ply terminations and closely-spaced tight-radius corners on thickness and porosity. Their results indicated that the quality of graded-thickness sections is optimal when each ply connects to the breather through at least one “breathing edge” or when the materials used have sufficient transverse permeability to enable air evacuation into adjacent plies. The study also indicated that thickness and void content depend on the consumable arrangement, and suggested that removing the breather from tight-radius areas is a simple, practical strategy for improved quality. A kinematic model was developed to understand the phenomena governing corner consolidation. Results from this model, along with experimental data, indicated that corner thickness variations are not simply a function of compaction or consumable bridging (which were accounted for in the model), but also resin migration from high-pressure flange areas to the lower pressure corner. Modifications to lay-up arrangements and cure cycles were also examined by Grunenfelder et al. [68] in panels with internal ply drop-offs (embedded doublers) and hat-stiffeners, emphasizing the need for adequate edge-breathing in VBO cure.

Teuscher et al. [69] reported on the quality of a T-shaped stringer part manufactured with VBO prepreg and three tool components: a flat base plate, and two triangular aluminum pressure intensifiers. Using this set-up, the authors reported thickness variations leading to fiber volume fraction values of 50% - 57%, with the lowest levels reported at the corner. To mitigate these defects, the authors tested an alternate tooling configuration that used shape memory wires to create additional pressure on the side moulds, and reported preliminary variability reductions of about 70%.

Finally, a parametric study was carried out by Bond and Luner on the influence of room temperature vacuum debulk cycles, bagging procedures and cure cycle on a discriminator panel

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containing ply drop-offs and variation in ply step size [70]. A design of experiments approach was utilized to examine the influence of debulk frequencies and times, vacuum levels and cure temperatures. Debulk temperature and frequency influenced void content in thick regions of the part, with elevated temperature steps yielding reduced part quality. Debulk parameters had no influence on thin areas. To examine bagging and cure cycle, two edge-breathing materials were used (cork dams and vacuum sealant tape wrapped in fiberglass cloth), and cure ramp rate, dwell temperature, cure time and vacuum level were varied. Vacuum level was the only statistically significant factor influencing void content, with voids increasing as pressure levels differed increasingly from perfect vacuum. Variations in cure time and temperature also influenced the glass transition temperature of the cured composite.

### *3.3.2 Honeycomb Structures*

VBO prepregs can be used as facesheets in honeycomb sandwich structures. In such cases, potential challenges arise not solely from geometric features, but also from air entrapped within the honeycomb cells, which can flow through the facesheets or, in the opposite case, remain entrapped and pressurize to dangerous levels during cure or subsequent part use. To save time and cost, composite facesheets can be simultaneously cured and bonded to the core with adhesive in a process known as co-cure (Figure 10A). VBO co-cure is attractive, as it eliminates the core crush that can occur under autoclave pressures, allowing for the use of lighter, less expensive cores [71,72]. Additionally, low-temperature, low-flow VBO resins can reduce resin flow into the core [73]. Vacuum co-cure can be problematic, however, as some adhesives out-gas under vacuum, leading to foaming during cure. Adhesive layers can also trap air within the core.

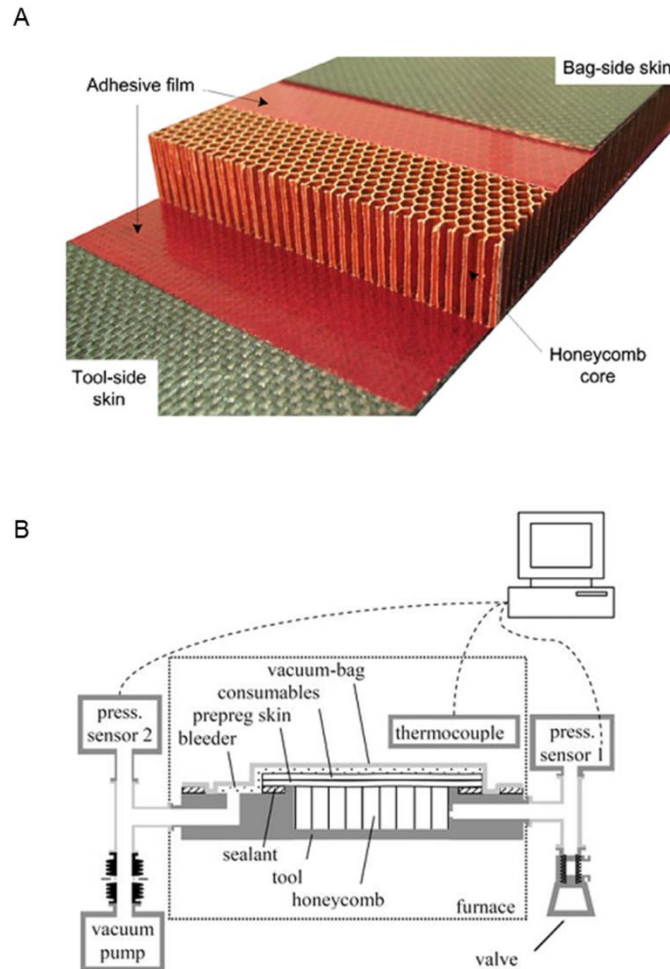
Several investigations have been performed on the influence of material properties and process parameters on air removal behavior and the feasibility of VBO co-cure, the results of which are Please cite this article as: Centea T, Grunenfelder LK, Nutt SR. **A review of out-of-autoclave prepregs - material properties, process phenomena and manufacturing considerations.** *Compos Part A Appl Sci Manuf* [Internet]. 2014; 70:132–154. Available from: <http://www.sciencedirect.com/science/article/pii/S1359835X14003108>





detailed below. Sequeira-Tavares et al. [43,71,74–76] carried out multiple studies on the VBO processing of honeycomb sandwich structures, with particular emphasis on the evacuation of core air through the prepreg facesheets. In a previously mentioned study, an instrumented method was developed and demonstrated for measuring the through-thickness prepreg permeability throughout processing (Figure 10B) [43]. The evolution of permeability was shown to be predictably complex. An initial decrease was observed with increasing temperature, a result of the dominant effect of prepreg impregnation over increased resin mobility. Next, a post-impregnation increase in permeability was observed, stemming from resin migration into the peel ply. Finally, permeability values stabilized at gelation. The authors also assessed the permeability of semipregs (or prepregs with alternating in-plane dry and resin-rich regions) [74]. Contrary to traditional VBO prepregs, such materials were highly permeable at the onset of cure and allowed easy core evacuation before becoming progressively less permeable during processing because of gradual impregnation. The low-viscosity resin in these systems, however, seeped into the honeycomb core cells, resulting in final parts with unacceptable dry areas. The authors thus proposed the use of semipreg materials in hybrid facesheets, combined with a material designed to prevent resin loss, such as prepreg. Such hybrid facesheet designs were investigated in a third study, and were shown to produce sandwich structures of comparable quality to panels with traditional prepreg skins [75].





**Figure 10:** (A) Cut-out of a honeycomb structure used in VBO prepreg co-cure. Reproduced with permission [44]. Elsevier, 2013. (B) Schematic of an instrumented fixture used to measure the flow of entrapped air and the permeability of VBO prepreg skins during co-cure. Reproduced with permission [43]. Elsevier, 2009.

Means of improving transverse facesheet permeability have been investigated also [76]. Prepreg and adhesive plies were perforated using several methods to create artificial evacuation channels of various density and morphology. Facesheets were then cured over honeycomb core while measuring the core pressure. The adhesive used in the study was identified as the primary impediment to air



evacuation, though perforating the entire laminate (rather than individual prepreg or adhesive plies) provided the greatest improvement in facesheet permeability and honeycomb core pressure. Finally, the role of honeycomb core pressure on skin-core adhesion and skin porosity was examined [71]. Core pressure influenced both quality metrics in two ways: by affecting the consolidation pressure differential imparted to the skin, and by potentially inducing adhesive out-gassing, but in a non-linear manner. An optimal initial core pressure of 40% to 70% of atmospheric pressure was identified.

Kratz and colleagues [44,45,54,77–80] carried out a parallel set of investigations on air evacuation from honeycomb core and the impact of air removal on sandwich structure quality. As previously mentioned, the authors used a lab-scale fixture and measurement methods inspired by Sequeira-Tavares et al [43] to determine the permeability of VBO prepreg facesheets, and ascertained that fiber bed architectures with larger interconnected macro-pore networks allow more effective and consistent air evacuation [44,45]. The effect of introducing perforations and gaps within the facesheet was also investigated, showing that both approaches can dramatically increase transverse air permeability, although not without some cost to mechanical properties [54].

The influence of the core air behavior on part quality was also studied by manufacturing full-size panels in conditions similar to those measured in the lab-scale fixture [77]. Prepreg facesheets that prevented core air evacuation prior to cure caused internal core pressures to reach and fluctuate around atmospheric pressure, resulting in panels with higher facesheet void contents and lower skin-core peel strength. Subsequently, the effectiveness of in-plane and through-thickness air evacuation was quantified by measuring core pressure in-situ in full-size sandwich panels using embedded core pressure sensors [78]. The transverse arrangement, being independent of panel size, removed a larger

quantity of core air more rapidly than the in-plane, and minimized pressure gradients within the

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sandwich, but led to variations in core pressure during cure because of the complex evolution of the through-thickness permeability. Conversely, the core pressure in the in-plane panel was greater but steady. The transverse panel facesheets exhibited more facesheet porosity and remained permeable once cured, while the in-plane did not. However, both panels showed comparable skin-core bond strengths, as measured by a climbing drum peel test.

The influence of core material properties on sandwich structure quality was investigated by exposing aluminum and Nomex honeycomb cores to various ambient humidity levels and manufacturing representative structures [79]. While the metallic core did not undergo any weight changes during conditioning, the Nomex core absorbed moisture in proportion to the relative humidity, and released it as vapor during processing. Because of the limited air evacuation capacity of the prepreg skin, this release led to a significant increase in core pressure. Despite these factors, however, all manufactured Nomex panels exhibited comparable facesheet void contents as a result of the relatively thin cross-section, which did not entrap significant air. Furthermore, for both aluminum and honeycomb core panels, all flatwise tension tests exhibited core failure. Finally, the authors developed and validated an analytical model describing the instantaneous honeycomb core pressure as a function of the initial pressure, the facesheet permeability during cure, the core moisture content, and the cure cycle [80]. The model, which combined flow through porous media and diffusion equations, captured experimental trends, and the authors showed that it could be used to predict core pressure for a variety of different cure scenarios given the required input parameters for the materials used.

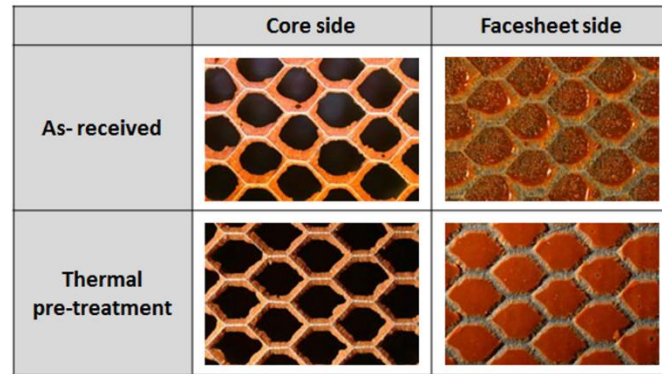
In addition to the significant work on prepreg facesheet behavior outlined above, studies have been carried out on the properties of the adhesives used in sandwich structures. Storage et al. [81]

screened multiple commercially-available adhesive systems to ascertain compatibility with VBO  
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temperature and vacuum process conditions. Adhesive properties were characterized by DSC, RDA and TGA, and facesheet-core specimens were manufactured and tested under ambient and hot/wet conditions. VBO cure generally resulted in higher void contents at the bondline, attributed to cure-induced volatile release from the adhesive (or “foaming”) under vacuum conditions. These defects were associated with a decrease in bondline shear strength, but other mechanical performance metrics remained unaffected. Overall, candidate adhesives displayed different advantages and drawbacks, and no single adhesive was deemed clearly superior over the full spectrum of requirements. In a follow-up study [82], two additional adhesive systems were examined.

Hou et al. [83] investigated a range of prepreg materials (IM7/MTM45-1 and T40-880B/5320) and adhesives (AF-555M, XMTA-241/PM15, FM-309-1M and FM-300K) for VBO co-cure of sandwich structures with aluminum cores. Thermal pre-treatments were applied to the adhesive films to reduce foaming by advancing the degree of cure of the material. While these pre-treatments reduced foaming in select adhesives (Figure 11), they also led to increases in minimum viscosity during processing, and lower flatwise tensile strength as a result of insufficient surface wetting. A moderate initial viscosity of 200 to 400 Pa·s was suggested as an optimal balance between surface wetting and high degrees of foaming, leading to desirable flatwise tensile strength values. Additionally, a significant increase in strength (31%) was observed with the same adhesive and facesheet materials when the core material was perforated.



*Figure 11: Reduction in foaming of adhesives during VBO co-cure following a thermal pre-treatment. Reproduced with permission [83]. Society for the Advancement of Material and Process Engineering (SAMPE), 2010.*

Chiou and Oldroyd [84] reported on VBO processing with various sandwich structure adhesives, as well as a range of honeycomb core materials, including high-performance cores made from glass and carbon fiber prepregs. Their observations showed that panels made with a core material that had larger cell size and thinner cell walls resulted in increased porosity on the bottom facesheet, as vacuum bag pressure could only be transferred through the cell wall contact areas. The pressure increase at the contact points resulted in a pinching off of pathways for air removal and thus increased porosity. VBO samples produced with prepreg cores showed increased flange thickness and reduced flatwise tensile strength when compared to autoclave cured parts, while no mechanical property difference was observed with traditional core materials.

Finally, Butukuri et al. [85] carried out manufacturing trials, flatwise tensile testing, and microstructural analysis of the effect of adhesive layer thickness, core material, and cell size on skin-core bond quality. Furthermore, they investigated the importance of reticulating the adhesive, or distributing it preferentially on the network of cell walls, using a targeted compressed air jet. Their

results demonstrated that thicker adhesive layers provided better mechanical performance, and that

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reticulation improved fillet size and bond strength for configurations in which the adhesive bondline is weaker than the core. Smaller core cells were shown to improve quality by increasing the available bond surface per unit area.

Mechanical property equivalence between VBO and autoclave processed sandwich panels has been explored in multiple studies. Coupons from sandwich panels produced using out-of-autoclave (T40-800b/5320-1) and autoclave (IM7/977-3) prepregs were tested by NASA to compare compression and compression-after-impact (CAI) behavior [86]. The panels showed statistically similar performance when tested in dry conditions at room temperature, as well as at elevated temperature and after hot/wet conditioning. In a similar study, Nettles and Jackson [87] reported on the CAI residual strength of three composite systems. Two out-of-autoclave (IM7/MTM-45 and T800/5320) prepregs and one autoclave (IM7/8552) material system were tested in impact after being bonded to an aluminum core. The impacted sandwich structures were then subjected to a three-point bend test to measure damage tolerance, with no major differences observed for the three systems. The largest deviation was observed at low impact energy in the IM7/MTM-45 system, which had the highest void content of the laminates studied.

The available literature on VBO processing of honeycomb sandwich structures is perhaps the most difficult to synthesize, owing to the large number of relevant parameters and the complexity and variability of their interactions. Several key points, however, do emerge. The permeability of the prepreg facesheets, which depends on the fiber bed architecture, evolution of resin viscosity, and laminate layup, should ideally allow sufficient flow to reduce the core pressure to between 33% and 66% of atmospheric pressure, so as to maximize the skin-core bond quality and reduce the potential for in-service pressurization and delamination without completely eliminating the facesheet

consolidation pressure differential. However, depending on the timing of air evacuation and the

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laminate properties, this air flow may degrade the microstructural quality of the facesheet itself. If an organic core is used, exposure to humid conditions should be limited to avoid moisture absorption and subsequent vaporization during cure. Furthermore, adhesives with limited volatile release at sub-ambient pressure are recommended to maximize bondline quality.

### *3.3.3 Demonstrators*

Demonstrator parts increase the level of complexity by an additional degree by combining large curvatures, sharp corners, ply drop-offs, honeycomb core segments, and other geometric features. Many such demonstrator parts have been fabricated during the programs used to develop VBO manufacturing technology. These programs are summarized in Table 3, and details of the research performed as part of each program is described below, along with a variety of independent research efforts.





**Table 3: Programs referenced in this work involving development of VBO prepreg technology and manufacturing**

PROGRAM	FUNDING BODY	GOALS	RELEVANT PARTIES	VBO PREPREG SYSTEM(S)	REF S.
Non-Autoclave (Prepreg) Manufacturing Technology	United States Defence Advanced Research Projects Agency	Enable pervasive, disruptive use of VBO prepreg systems for primary structure	United States Air Force, Boeing, Cytec	CYCOM 5320, CYCOM 5320-1	[9,12,66,70,73,93,94]
Lightweight Space Structures and Materials / Composites for Exploration (CoEx)	National Aeronautics and Space Administration (NASA)	Develop a composite heavy lift launch vehicle with a lift capacity of 70-100 mT	NASA	CYCOM 5320-1	[86,95]
Advanced Composite Cargo Aircraft (ACCA)	United States Department of Defense (DoD), Air Force Research Laboratory (AFRL)	Demonstrate advanced composite structure concepts, design and manufacturing to design, build and fly a primarily composite aircraft	Lockheed Martin, ACG	ACG MTM-45	[96]
Low temperature cure, cost effective composite materials for aerospace structures using out-of-autoclave processing (EFFICOMP)	UK Government	Development of out-of-autoclave resin systems for cure at 80-90° C, production of honeycomb sandwich panels, use of heated tooling	ACG, GE, BAES, GKN Aerospace, Sigmalex		[97]
Integrated Wing, Advanced Technology Verification Programme (IW-ATVP)	UK Department of Trade and Industry (DTI), Regional Development Agency (RDA)	Work Package 2 (structures): Production, out-of-autoclave, of a composite wing leading edge for bird strike evaluation	Airbus, Bombardier Aerospace, GE, ACG	ACG MTM 44-1	[97]

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Hughes and Hubert carried out a systematic investigation using VBO cure to directly replace a common autoclave cured part in a case study examining scale-up for out-of-autoclave processing [88]. The study progressed in complexity, beginning with L-shaped brackets and demonstrator coupons to optimize layup methods, and finally moving up to a full-scale part (Figure 12A). The complexity of the full part, which contained multiple ply drop-offs, curvatures, and lack of ply alignment at the edges of the part, made edge breathing impractical. Voids associated with ply drop-offs were observed in the final part, and void content increased with part size owing to the larger distances required for air removal.

Lane et al. [89] described the design and manufacture of a 2.8 m-diameter space launch vehicle fairing featuring large-curvature and conic sandwich sections. While few details were provided on the prepreg materials and layup specifics, cured part void contents of less than 2% were reported, suggesting that OOA/VBO prepreps were used to manufacture the large curved structure with acceptable quality.

In 2007, a team at Boeing, under the guidance of the United States Air Force and the Defence Advanced Research Projects Agency (DARPA), initiated a research program on out-of-autoclave technology. This “Non-Autoclave Manufacturing Technology Program,” along with other efforts at Boeing, led to the production of a range of demonstrator parts with increasing complexity. The goal of the work was to move from manufacture of tertiary and secondary structures to VBO production of load-bearing and primary structural components. In early publications in this effort, Bernetich detailed the manufacture of a VBO fairing cover, demonstrating the drapability of VBO prepreps over complex curves [90]. An aircraft cabin frame was also produced in this work, using both VBO and autoclave methods. The two parts were shown to be equivalent, with void contents less than 0.5



% . Finally, parts with integral ply drops were produced with out-of-autoclave prepregs, yielding less than 1 % porosity.

In another study, the performance of a VBO-processed part (5320/T40-800B) was evaluated in a one-to-one comparison with a well-qualified autoclave baseline (IM7/8552) [91]. Coupons from various regions of each part were removed and subjected to a range of tests, including open-hole compression, short beam shear, combined loading and compression, and tension and flange bending. Resin content and glass transition temperature were also measured. The resin content in the flange of the VBO processed part was low, but mechanical testing showed autoclave equivalence. Laminate quality, assessed through examination of polished sections, also showed autoclave equivalence, with no observable voids, resin-rich areas, or ply wrinkling.

Further scale-up efforts compared autoclave and VBO co-cured skin-stiffener bonded assemblies (Figure 12B) [92]. Impact damage was imparted to the samples in two locations and ultrasonically inspected. Subsequent static and fatigue tests were carried out under ambient and low-temperature (-54° C) conditions. The same lay-up consumables, mandrels, and caul plates were used for both autoclave and out-of-autoclave cure, with the only difference being edge-breathing protocols for the VBO samples. When compared to autoclave cured parts, the VBO-processed samples required greater or equal impact energies to show visible damage, passed all part load level requirements, and failed at equivalent or higher loads under all conditions tested. Both unidirectional prepregs and woven materials were tested.

As part of the same research effort on the scale-up of VBO processing, Boeing produced several demonstrator parts, including a co-cured torque box and co-cured aft cooling duct, both with complex geometries [73]. Additionally, 21-m wing skins of various configurations were fabricated and tested (Figure 12C) [12]. These configurations, which achieved aerospace quality, included

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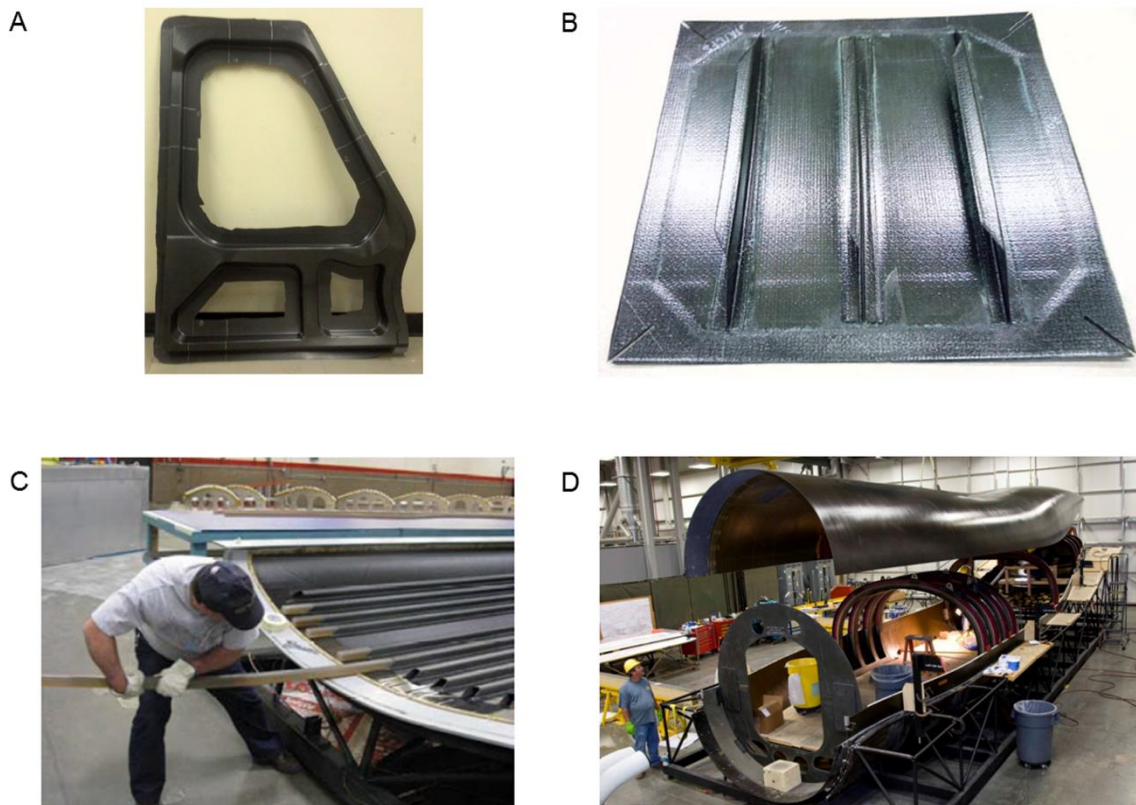
plank-stiffened (monolithic), hat-stiffened [93], and core-stiffened structures. Bond et al. [94] reported on the manufacturing of complex, 5.2 m-long, drape-formed spars, with results provided from a part made primarily of high-modulus fiber, hand-laid prepreg and localized honeycomb core inserts. Some quality issues were identified, including surface porosity above the core sections and wrinkles at corners from laminate “buckling.” Porosity levels varied between acceptable (1% to 2 %) in flat areas, to 5% in the corner. The authors concluded that these results, while preliminary, indicated that despite minor issues, drape-forming of OOA/VBO prepreg structures was feasible for complex aerospace structures.

In a separate research effort, 1/16<sup>th</sup> arc segments of a 10 m diameter cylindrical payload fairing for a launch vehicle were manufactured both in- and out-of-autoclave by NASA under the Lightweight Space Structures and Materials program [95]. The panels consisted of an aluminum core and 8-ply quasi-isotropic graphite/epoxy facesheets, fabricated using an automated tape-laying machine. In tension and compression coupon tests, the VBO-processed face sheets showed greater strength, particularly under hot/wet conditions, but lower stiffness, than the autoclave-cured material. Similar trends were observed in small-panel tests, with VBO samples being stronger and more compliant, a result attributed to the stronger and tougher matrix in the VBO prepreg system. The buckling response of the VBO and autoclave systems was comparable. As a result of the reduced compaction pressure and lack of a caul plate, however, the VBO processed demonstrators were more susceptible to cosmetic imperfections, as well as to bulging caused by core splice regions, which has been shown to reduce strength.

The Air Force Research Laboratory utilized VBO prepregs to produce the fuselage of its Advanced Composite Cargo Aircraft (ACCA) [96]. The fuselage measured 19.8 m from nose to tail, and the sandwich stiffened fuselage skins (Figure 12D) along with the frames, floor supports, Please cite this article as: Centea T, Grunenfelder LK, Nutt SR. **A review of out-of-autoclave prepregs - material properties, process phenomena and manufacturing considerations.** *Compos Part A Appl Sci Manuf [Internet]*. 2014; 70:132–154. Available from: <http://www.sciencedirect.com/science/article/pii/S1359835X14003108>



fairings, the cargo door, and the pressure bulkhead, were all fabricated with VBO prepreg (MTM-45). The finished structure underwent a full-scale proof test, showing structural integrity when subjected to 100% design limit loads for fuselage bending. VBO prepregs have also been used in the fabrication of wind turbine blades, racing yachts, and prototype submarine structures [21], as well as in wing structure components incorporating ply drops and honeycomb core [97].



**Figure 12:** Demonstrator parts made from VBO prepregs: (A) full-scale part with ply drop-offs and complex curvature. Reproduced with permission [88]. Hughes and Hubert, 2013; (B) Co-cured skin-stiffener bonded assembly. Reproduced with permission [92]. Society for the Advancement of Material and Process Engineering, 2010; (C) Wing skin demonstrators. Reproduced with permission [12]. Society for the Advancement of Material and Process Engineering, 2011; (D) Fuselage skins (upper and lower). Reproduced with permission [96]. Society for the Advancement of Material and Process Engineering, 2009.



The studies discussed above have shown that complex geometric features can present challenges for VBO cure because of impeded air evacuation and/or reduced pressure for laminate forming. However, the reviewed studies have also identified feasible defect-reduction strategies and guidelines based on consumable arrangement or tooling adjustments, and several large-scale demonstrator manufacturing trials have confirmed that complex parts of acceptable quality can be manufactured using VBO methods.

The research programs discussed above and listed in Table 3 have primarily been research efforts between airframe and material manufacturers, usually located in the United States and United Kingdom, and directly associated with the development and scale-up of VBO prepreg processing. However, it is worth noting that, in recent years, significant other efforts to further advance or take advantage of OoA manufacturing have been underway. For example, the European Commission Seventh Framework Programme (FP7) included two projects, focused on heated tooling for out-of-autoclave composites (“Industrialization of Out-of-Autoclave Manufacturing for Integrated Aerostructures”) and out-of-autoclave repair (“Innovative Repair of Aerospace Structures with Curing Optimization and Life Cycle Monitoring Abilities”). Similarly, the Clean Sky Initiative, a research partnership between the European Commission and Industry, is currently encouraging research on the out-of-autoclave manufacturing of primary structures (“Out-of-Autoclave Composite Manufacturing, Wing and Tail Unit Components and Multifunctional Design”). Finally, the G8 Research Councils are currently funding a multi-national research effort on “Sustainable Manufacturing Using Out-of-Autoclave Methods” between academic institutions in the United Kingdom, Germany, Canada and the United States (including the authors’ institution), under the guidance of an international industrial advisory board. Within this context, the summary of major

research programs provided in Table 3 and in these paragraphs is by no means exhaustive, but Please cite this article as: Centea T, Grunenfelder LK, Nutt SR. **A review of out-of-autoclave prepregs - material properties, process phenomena and manufacturing considerations.** *Compos Part A Appl Sci Manuf* [Internet]. 2014; 70:132–154. Available from: <http://www.sciencedirect.com/science/article/pii/S1359835X14003108>





indicates that interest in VBO prepregs and OoA methods is intense, and provides starting points for further research by the interested reader.

### 3.4 Cost and Environmental Considerations

While VBO prepreg processing is often promoted as a comparatively cost-efficient and environmentally friendly alternative to autoclave cure, few published studies have specifically carried out cost and life cycle assessments (LCAs). These studies are reviewed and discussed below to assess VBO performance.

Ridgard [4,6] discussed manufacturing costs while motivating the need for VBO prepregs, and explained that low-temperature (and low-cost) tooling is a major advantage, particularly for low production runs, in which material costs for high-temperature tools can account for up to 70% of the total cost. Furthermore, he argued that VBO cure can potentially lead to improved part accuracy and reduce post-cure machining and assembly times because of reduced thermal expansion effects. Finally, he specified that lower cost tooling and non-autoclave cure could enable the manufacturing of larger integrated structures, which can reduce assembly cost and time.

Boyd and Maskell [7] discussed various emerging technologies for low-cost manufacturing, including VBO prepregs, emphasizing that low-pressure processes may reduce scrap rates by mitigating autoclave-induced defects such as honeycomb core crush and facesheet dimpling. They further noted that non-autoclave processing can eliminate the high capital and operating costs of an autoclave and allow the integral manufacturing of large parts that would otherwise require expensive and time-consuming assembly.

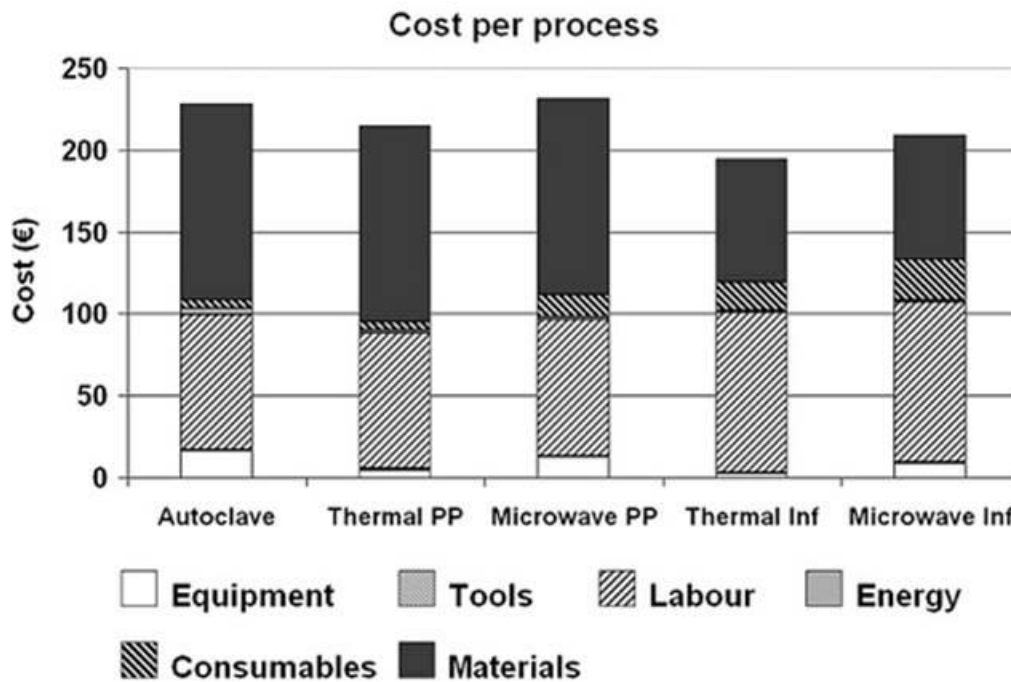
Witik et al. [98] carried out a detailed economic and environmental analysis of several production methods for a  $400 \times 400 \times 4$  mm representative flat aircraft component, comparing autoclave prepreg

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cure to VBO prepreg and resin infusion in conventional and microwave ovens. Technical cost modeling showed that for the part and manufacturing approaches studied, raw materials and labor account for a large majority of costs, with contributions from equipment, tooling, energy and consumables being comparatively small (Figure 13). VBO oven cure was predicted to reduce the cost per part by 6% relative to the autoclave, primarily because of a large reduction in capital equipment costs and subsequent energy savings. A life cycle assessment using the IMPACT 2002+ method also indicated that VBO prepreg cure improves on autoclave cure in several environmental performance metrics (greenhouse gas emissions, resource use, ecosystem quality and human health) by between 10% and 20% (as measured using each category's appropriate unit), primarily through reductions in energy consumption. However, as with cost, the manufacture of the raw materials (particularly the carbon fibers) comprises a large fraction of the total energy usage. Interestingly, while all non-autoclave alternatives showed overall improvements over the autoclave benchmark, the resin infusion process performed better than VBO prepregs both economically and environmentally owing to the lower cost and reduced environmental burden involved in producing the base materials (dry fiber and neat resin versus prepreg). However, the technical suitability of each method for aerospace structural components and the expected quality and consistency of manufactured parts was not compared.



*Figure 13: Comparison of the manufacturing costs of a generic composite laminate for autoclave, thermal and microwave oven prepreg (PP), and thermal and microwave oven vacuum infusion (inf) processes. Reproduced with permission [98]. Elsevier 2012.*

Teuscher et al. [69] used the same framework to compare autoclave and VBO prepreg manufacturing of ribbed parts. Conclusions of this study were similar: while oven cure provides a cost and environmental impact reduction by means of lower energy consumption and cycle time reductions, the gain is limited by the large fraction of the total cost associated with material costs.

The above studies indicate that VBO prepreps provide an incremental economic and environmental benefit, in addition to the elimination of the technical constraints associated with autoclaves. The decision to begin or transition to VBO prepreg manufacturing will likely depend on a combination of technical, economic and environmental factors, as well as on existing infrastructure, previously qualified materials, institutional expertise and production rate

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requirements. For companies possessing autoclaves and qualified materials, a complete transition to VBO cure may not be justifiable unless the autoclave significantly limits throughput, or if very large parts must be manufactured. Conversely, companies seeking to enter the composites field may find VBO prepregs useful in lowering technical and economic entry barriers.

## 4. Present Challenges

### 4.1 Material and Process Sensitivity

While autoclave-quality parts have been produced using vacuum bag-only prepregs, there are unique features inherent to VBO methods that require careful consideration. VBO processing is a departure from autoclave methods, where manufacturers rely on high applied pressures to compensate for errors and imperfections in lay-up and material factors, such as absorbed moisture, air entrapment and out-time. Successful vacuum bag cure, therefore, requires careful material storage and handling, along with a skilled workforce and careful attention to lay-up procedures. Additionally, the engineered vacuum channels that make vacuum bag cure possible introduce issues in terms of bulk factor. Since each prepreg ply is initially only partially impregnated, a laminate undergoes significant compaction during cure. This large thickness change from the as-laid-up state can lead to wrinkling and fiber bridging in contoured parts. This is particularly significant in woven fabric preforms, which have a higher initial bulk factor than unidirectional prepregs [28].

In addition to the complexities of producing large or contoured parts using out-of-autoclave prepregs, the introduction of new materials into industrial manufacturing poses considerable challenges. Typically, numerous production runs are required to qualify a material for aerospace use.



Additionally, the process by which accurate and repeatable performance of a material is demonstrated for production-scale parts is time-consuming and costly, typically spanning 15-20 years, and requiring thousands of tests and millions of dollars of investment [99]. Large portions of development time and resources are commonly spent fixing unanticipated problems that arise during iterative research and development efforts (Figure 14A) [100,101]. This leads to a risk-adverse outlook, with part manufacturers relying on proven materials and methods, reluctant to adopt new materials and designs. To address these issues, past efforts have been undertaken with the aim of accelerating the insertion of materials into hardware, notably the Department of Defense and National Research Council Committee on Accelerating Technology Transition [102], and a DARPA initiative focused specifically on fiber-reinforced composites for defence applications, entitled Accelerated Insertion of Materials – Composites (AIM-C) [103]. The goal of these programs was to utilize simulation tools, uncertainty analysis, and intelligent test protocols to speed the introduction of new materials into critical components.

A required element for accelerated insertion efforts is robust material and design databases. One such knowledge database was developed and incorporated into the AIM-C Comprehensive Analysis Tool (CAT), along with experience-based heuristic models and scientifically based analytical models (Figure 14B) [99,100]. The AIM-C CAT approach resulted in a demonstrated reduction in the time and cost of materials insertion. For VBO materials, steps have been taken to create similar resources by NCAMP, the National Center for Advanced Materials Performance [104]. The purpose of NCAMP is to produce shared material databases to aid in the qualification and use of new materials. Thus far, three VBO prepreg resin systems have been added to public databases, with a range of preform types (see Table 4).

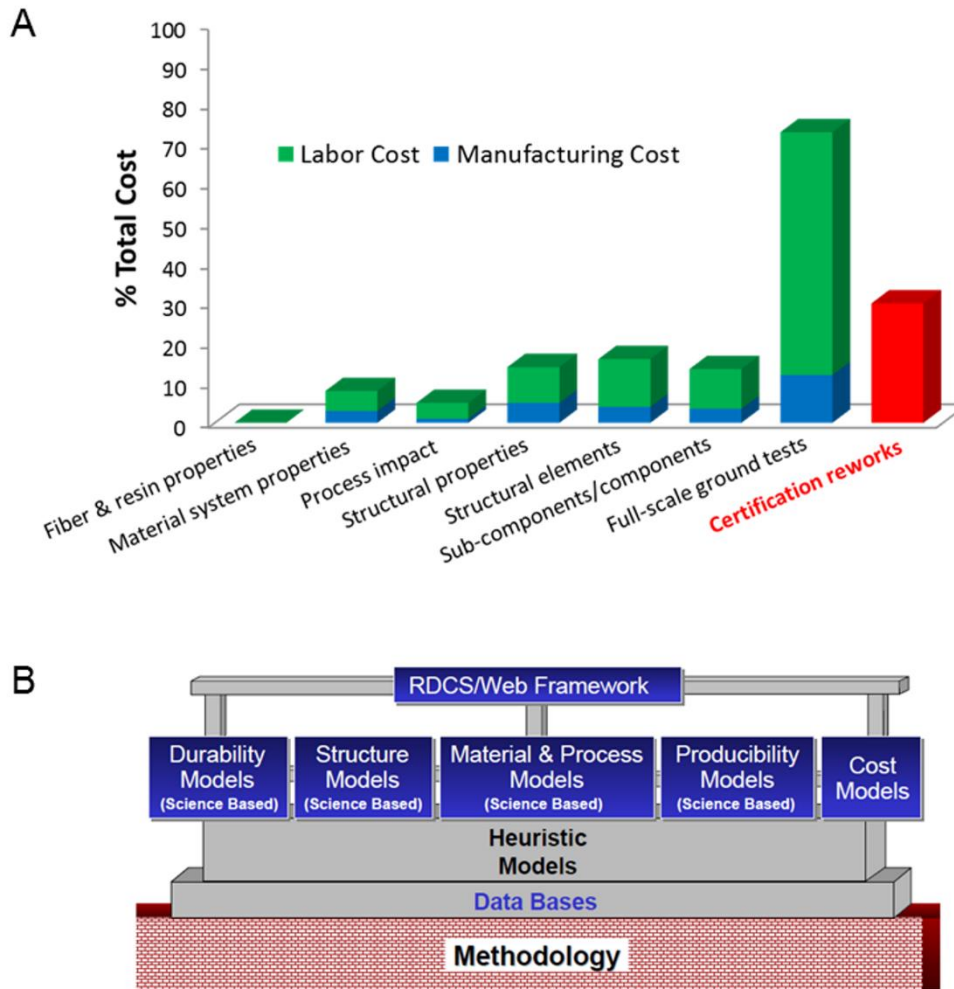
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**Table 4:** Programs referenced in this work involving development of VBO prepreg technology and manufacturing

Resin System	Preform Types
MTM45-1	12K IM7 Unidirectional
	6781 S2 Glass
	3K G30-500 Fabric Plain Weave
	AS4-145 Unidirectional
	6K 5H AS4C Fabric
	CF0525 3K PW AS4 Fabric
CYCOM 5320-1	T650 Plain Weave
	T650 Unidirectional Tape
TenCate TG250	Not yet available

The bulk of VBO processing to this point, as clear from the above discussions, has focused on toughened epoxy resin systems. Other resin systems, however, including polyimide [105], bismaleimide [106], benzoxazine [107,108], and cyanate ester (see Table 1), have been formulated with flow profiles and reactivity suitable for out-of-autoclave manufacture. These materials open up the design space for a range of processing and service temperatures, storage and service environments, and mechanical properties.



**Figure 14:** (A) Example of cost associated with the development of a composite part, with reworks accounting for more of the total cost than constituent, coupon, element, sub-component and component testing combined. Adapted from [101]; (B) Methodology of the AIM-C CAT tool, incorporating methodology, material databases, and heuristic and scientific models to accelerate the insertion of new materials into composite components. Reproduced with permission [100]. Society for the Advancement of Material and Process Engineering, 2002.



## 4.2 Scale-Up

A key factor in the scale-up of VBO processed parts to large structures is the ability to determine part quality through non-destructive test methods. This typically requires the manufacture of porosity standards (panels with controlled porosity levels) that can be used to correlate void content to ultrasound attenuation for each unique material system. Early in the production of VBO parts, porosity standards produced from autoclave curing systems were used, assuming that the relationships between porosity, ultrasonic attenuation and mechanical properties were similar for VBO materials. This assumption was tested through the development of porosity standards for Cytec's 5215 VBO material [109]. While large discrepancies were not observed between the autoclave baselines and VBO panels, each prepreg material has a unique formulation and will produce slightly different results. For this reason, porosity standards have been developed for the CYCOM 5320-1 material, with both unidirectional and fabric reinforcement [110]. Different vacuum levels were used to control porosity, and void content was determined through acid digestion. A trend of increasing attenuation with increasing void content (decreasing vacuum) was observed and used to establish a correlation between porosity and ultrasound attenuation. Similar efforts will likely be required for all VBO systems.

The integration of VBO prepreps into automated material deposition systems is also critical for large-scale parts and high-rate production. In aerospace, primary structures are often manufactured using automated fiber placement (AFP) and automated tape placement (ATP), which enable faster material laydown rates. Automated methods use unidirectional reinforcements as the base product form. However, so far, most fundamental research on VBO prepreps has focused on fabric systems, which are more often applicable to secondary structures. Several authors have emphasized the

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differences between typical materials for automated systems and VBO prepregs. For example, Lucas et al. [11] noted that AFP materials typically have higher degrees of impregnation than those used in hand layup. However, AFP placement is also assumed to mitigate void formation due to the pressure and heat applied locally by the head. Steele et al. [16] elaborated by explaining that, for AFP techniques, it is difficult to ensure air removal without fully-impregnated UD tapes, because of the complex nature of the placement head, the requirement for low bulk factor tapes, and the fact that not all tape courses may connect to an edge breathing dam. However, they also explained that the product forms used in automated processes are actually less likely to entrap air than during hand layup, if used correctly. The authors also noted that while some ply adhesion is essential during hand layup, automated methods generally require less tack to prevent air entrapment. The ability to produce high quality parts using ATP with vacuum cure has been demonstrated by NASA during the manufacturing of components for the Heavy Lift – Space Launch System (HL-SLS) payload shroud [86,95]. However, as emphasized in this review, successful VBO processing is highly dependent on appropriate combinations of material, part and process factors, and requires a detailed understanding of the underlying physical phenomena. An improved understanding of the unique consolidation and defect formation mechanisms associated with automated placement methods is desirable to ensure the robustness of automated VBO manufacturing.

## 5. Conclusions

VBO prepregs allow the manufacture of autoclave-quality parts under vacuum bag-only compaction, using conventional ovens or other non-autoclave setups. This class of materials feature a partially impregnated microstructure that promotes gas evacuation and suppresses defect formation

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in the initial stages of processing before being fully saturated with surrounding resin during cure. The coupled air evacuation, fiber bed compaction, resin flow and void growth (or collapse) phenomena that constitute consolidation/compaction depend on a combination of factors, including the properties of the constituent fibers and resin, the initial state of the prepreg, cure parameters such as temperature, vacuum quality and consumable arrangement, and part characteristics such as geometric complexity and size. These relationships have been investigated in multiple studies, demonstrating that high quality parts can be successfully fabricated under appropriate conditions. However, these studies have also demonstrated that VBO prepreg processing can result in unacceptable quality if key parameters deviate from their allowable range. OoA processing reduces the costs and environmental impacts relative to traditional methods by decreasing energy consumption during cure. However, additional research and development is required to improve process robustness and fully optimize the scale-up of VBO processing to industrial production levels.

The broader impact of VBO prepregs within the composites industry, and their standing as “hype or revolution” will only become apparent with time. However, out-of-autoclave processing represents a larger paradigm shift for advanced composites manufacturing, with VBO prepregs providing an initial step out of the autoclave. The first few decades of commercial VBO composites use focused primarily on developing materials and methods to achieve the desired quality, performance and reliability targets. At present, the attention of the community is shifting to strategies for faster, more efficient, and environmentally benign manufacturing. VBO prepregs both embody and enable this shift. They allow high performance structures, which previously required expensive and complex equipment, to be manufactured in a variety of environments with lower-cost infrastructure and tooling. Nevertheless, they use many of the traditional autoclave techniques and practices, eliminating the need for advanced technician training or development of specialized

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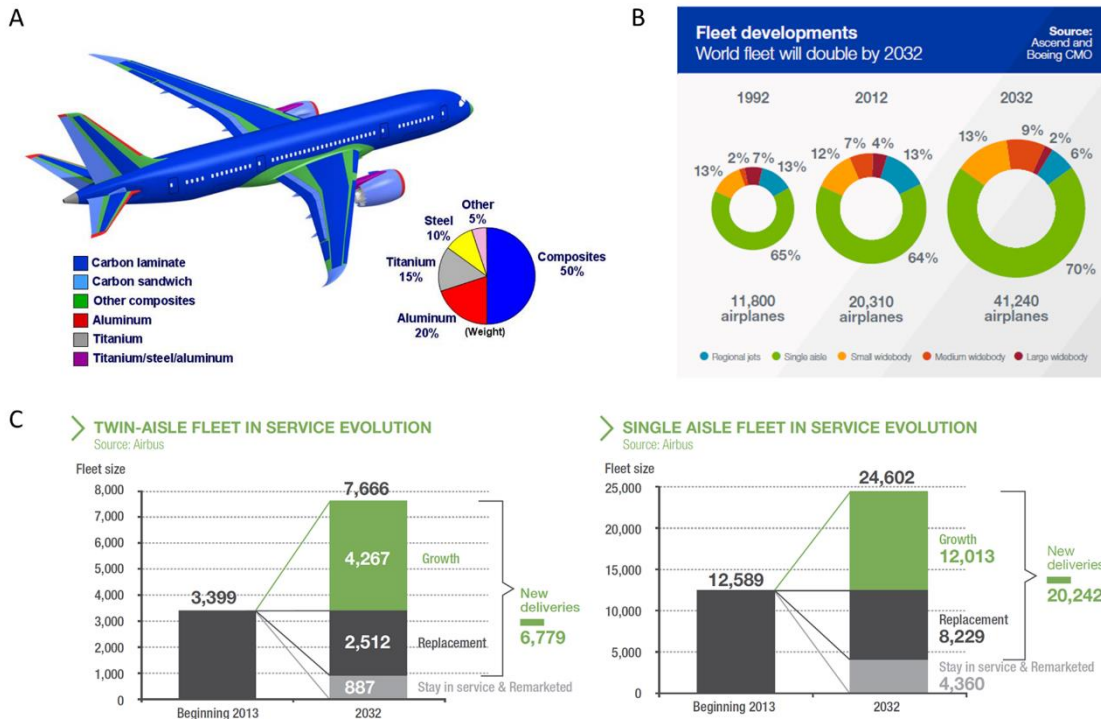
equipment. For aerospace and similar high value applications, VBO prepregs enable the installation of parallel production lines, in which one autoclave is replaced by several oven-based curing environments. In the aerospace industry, efforts are underway to replace metallic components with unitized composite structures. The predominant example of this trend is the Boeing 787 Dreamliner, whose airframe consists of 50% composite materials by weight (Figure 15A), though other aircraft currently in development or certification (such as the Airbus A350 XWB, Bombardier C-Series and Boeing 777X) will include major composite primary structures. The manufacturing technologies used to produce these composite airframes are well-suited to wide-body models (like the 787), for which projected production demands are relatively low (Figure 15B). Conversely, projected demands for single-aisle (or narrow-body) aircraft are more than three times greater (Figure 15C), and the production rates required to meet this demand will not be possible with autoclave processing. Alternative manufacturing methods will therefore be required to transition single-aisle aircraft to composite-based designs. VBO processing is one such alternative. In addition, for other industries, the ability to produce composites without an autoclave may significantly reduce the economic entry barriers associated with purchasing and operating autoclaves, and allow composite materials to be considered for applications typically restricted to traditional, less expensive metals and plastics.

By significantly expanding the design and processing possibilities of high performance composites, VBO prepregs enable other technologies previously unattainable with the constraints of autoclave cure. In the absence of above-atmospheric pressures, materials can be cured on lower cost tooling – or, conversely, on heated tools that render even ovens unnecessary. Honeycomb structures may benefit from lighter and cheaper sandwich cores, which are less likely to crush under vacuum consolidation. Additionally, in-situ scarf repairs with high microstructural quality are possible with



OoA techniques. These, and other likely developments, further increase the number of possible process configurations.

For completeness it is worth noting that in the coming years, VBO prepregs are likely to be one of several technologies contributing to the paradigm shift towards rapid, efficient and increasingly sustainable processing. A range of non-autoclave methods currently exists, including resin transfer molding, vacuum infusion, and pultrusion. Each method has distinct advantages and limitations, and is best-suited to specific material types, structural geometries, part quality and performance demands, cost structures, and industries. With increasing manufacturing options and rising part counts, a detailed understanding of the fundamental science and applied manufacturing characteristics of each approach will be essential for their appropriate, optimized and ultimately successful use. Clearly, meeting the growing demands for composites will require a departure from traditional autoclave methods, and VBO processing is a viable step in that direction.



**Figure 15:** (A) Schematic of the Boeing 787 Dreamliner, a double aisle aircraft composed of 50% composite by weight. Reproduced with permission [108]. Society for the Advancement of Material and Process Engineering, 2008; (B) Projected demand for aircraft between 2013 and 2032. Reproduced with permission [109]. Boeing, 2013. (C) Airbus projected demand for twin-aisle vs. single-aisle aircraft.



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