Technical Paper

Processing and Characterization of Pultruded Polyurethane Composites

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Abstract

During the past five years, several new resin systems and process methods have been commercialized in the pultrusion industry including hybrid unsaturated polyester-urethane (UPE-PU) and two-component polyurethane (PU) resins. The push for new resins and process technology has been driven by pultruders seeking improved performance over traditional resins such as unsaturated polyesters (UPE) and vinyl esters (VE) and opportunities to develop new applications. Two-component polyurethanes are now widely known to exhibit superior strength and toughness and may allow manufacturers to cost-effectively produce lighter, stronger and more damage tolerant profiles. However, translating this improvement in performance into component design and process parameters is less well understood in the industry. This paper will provide a practical discussion of injection box design, material handling, component metering equipment, die design, and process conditions, aiding component manufacturers in producing polyurethane profiles in a robust pultrusion process. In addition, physical properties of PU profiles will be compared to UPE, VE and hybrid UPE-PU resin profiles with identical reinforcements. Mechanical and impact properties, resistance to environmental exposure and performance in secondary operations such as attachment strength will be described for each resin system.
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Introduction

The pultrusion industry is one which thrives on innovation. Pultruders, design engineers and composite end-users are always seeking to differentiate their products in order to expand into new market segments. One means to do so involves introduction of ever higher performing matrix materials. Historically, however, material performance has been inversely proportional to productivity in the pultrusion industry. That is, higher performing resins such as epoxies and vinyl esters are more difficult to pultrude than unsaturated polyester resins and do so at much lower line speeds.

In the past seven years, polyurethane resins have drawn considerable interest as composite matrix materials, especially for pultrusion processing (Ref. 1-8). This interest stems from the outstanding composite properties and potential for high pultrusion line speeds that have been reported using polyurethane matrices. Additional appeal stems from the high transverse strength, interlaminar shear strength and damage resistance exhibited by polyurethane composites which create the potential for simplifying the reinforcement lay-up and reducing profile thickness. In a break with the paradigm of pultrusion resins, the property advantages of pultruded polyurethane composites are not usually compromised by limited productivity due to low line speeds or high pull forces. The property and, in some cases, process advantages offered by polyurethane profiles are key factors in achieving the product differentiation which pultruders seek.

A substantial body of knowledge has been developed to address processing, performance and cost of pultruded polyurethane composites over the past seven years. This paper will discuss guidelines for component metering equipment, injection box design and process conditions to provide pultruders with a starting point to produce polyurethane profiles in a robust pultrusion process. In addition, physical properties of PU profiles will be compared to unsaturated polyester (UPE), vinyl ester (VE) and unsaturated polyester-urethane hybrid resin profiles with identical fiberglass reinforcement. Mechanical properties, properties after environmental exposure and performance for secondary operations such as attachment strength will also be described for each resin system. With this information in hand, composite design engineers, pultruders and end users will better understand how PU pultrusion technology can enable differentiated pultruded products through composite redesign, improved safety factors or reduced assembly costs.
Polyurethane Pultrusion Processing

As with any other new resin technology, there are unique material handling and process characteristics for polyurethane resins that must be considered to optimize composite part production. Early studies reported minimal details of specific differences between PU pultrusion and traditional resin systems. The PU processing knowledge base has grown to the point where full details of recommended metering equipment, injection box design, process conditions, material handling and environmental, health and safety (EHS) issues have been recently reported (Ref. 9). These recommendations are summarized in the following section. A schematic of a typical polyurethane pultrusion set-up is shown in Figure 1.

**Metering Equipment**

Polyurethane resins are two-component systems comprised of a polyol blend and an isocyanate, typically based on a modified polymeric MDI (diphenylmethane diisocyanate) such as SUPRASEC® 9700 isocyanate. The RIMLINE® SK 97007 polyol is a fully formulated blend of base resin, catalyst, internal mold release (IMR) and other additives. Pultruders have the option of modifying the resin by using additives such as filler, colorant or UV stabilizer.

To process this system, a two-component metering unit is required for production runs since the mixed resin has a limited pot-life (15-22 minutes depending on ambient temperature and mix quality). The metering unit dispenses mixed resin into a closed injection box or injection die. The required metering unit is comprised of several subsystems which are commercially available including metering pumps or cylinders, resin tanks, a mixhead and transfer hoses, mixing elements and a solvent flush system. Complete metering systems with a broad range of technical sophistication can be purchased from several commercial suppliers.

Photos of a typical metering unit and its components are shown in Figures 2 and 3. In this case, a dual-action air-driven cylinder pump is used to meter both the isocyanate and polyol from their day tanks to the mixhead where the resin is combined. Thorough mixing of the chemical components occurs downstream of the mixhead in static mixing tubes. The unreacted polyurethane resin then flows into an injection box where the composite reinforcement is wet-out and then subsequently pulled into the pultrusion die. A variety of additives and/or fillers may be included (typically in the polyol component) provided the component mix ratios are adjusted to maintain proper stoichiometry. The compatibility of such additives should be tested with the polyurethane system before processing.

It is necessary to flush the mixhead, mix tubes and injection box at the conclusion of a pultrusion run to prevent resin from curing in place. The flush solvent is typically introduced into the mixhead through a three-way ball valve. Resin is initially purged out of the mixhead and mix tubes with air followed by thorough flushing with solvent. Typically, a pressure pot works well as a solvent container. The solvent should have a high flashpoint to minimize the hazard of fire when passing it through a hot die.

**Injection Box**

Injection processing for pultrusion has been practiced in development and production for more than a decade. The approach reported by Gauchel & Lehman (ref. 10) using a ‘tear-drop’ injection die is probably the best known. Resin injection can be performed internally within the die, typically by modifying it with a weir surrounding the profile, or within an injection box added on to the die in place of a typical wet-bath. Each has its advantages. The configuration of the wet-out chamber for closed injection processes is considered to be highly proprietary to pultruders. However, there are several ‘rules-of-thumb’ that are known in the public domain.

The most critical design parameter is limiting ‘dead spots’ in the injection zone, especially for fast reacting resins such as polyurethanes, where the resin can accumulate and possibly cure. The entire volume of the injection box or die must be replenished 3-5 times before the gel time to ensure long runs can be made without gelation. Profiles can be fabricated with polyurethane resin using either rovings alone or a combination of mat and rovings. Each requires a different approach to injection box design,
particularly in constraining the reinforcement into the correct location and preventing leakage of resin from the injection box. In the case of both unidirectional and mat reinforced profiles, optimum wet-out is achieved through a combination of pressure and residence time in the injection box. The mixed polyurethane resin typically is injected through two or more ports to smooth out pressure and velocity gradients in the box or weir. The location and number of ports has a great impact on wetting efficiency.

**Die Design**

There are no special die design requirements for pultruding polyurethane resins compared to styrene-based resins. A typical die has a 3.2-mm (0.125-inch) inlet radius and has parallel walls along the full 0.9-1.0 meter (36-39 inch) length. Dies designed with an inlet taper may be used, but, as with all dies, entrance cooling is required to prevent cure at the inlet and subsequent die lock. Certain dies may have an exit taper. Such dies can be used as well, but may limit line speed if the gel zone shifts into the exit taper region, degrading surface quality. Polyurethane resins shrink considerably less than styrene resins, typically 2.0-2.5 % for the base resin and approximately 1.0% or less for the composite. Although there have been limited studies directly comparing lay-ups, pull forces for PU pultrusion have been found to be similar to UPE and VE despite the low shrinkage.

**Process Conditions**

There is some perception within the pultrusion industry that polyurethane resins are difficult to process and are prone to die lock. With a well formulated PU resin system and appropriate process conditions, polyurethanes exhibit quite robust and reproducible processing. High quality PU profiles have been run continuously for over 48-hours at 60-inches/minute using a standard polyurethane pultrusion resin. Using a well-designed closed injection box while dosing resin with a metering pump, runs of indefinite time are feasible.

As shown in Figure 4, the die should be set up with cooling at the entrance (~10-cm, 4-inches) to prevent heat from diffusing into the injection box, causing die lock. Production runs typically use two subsequent heating zones with Zone 1 at 160-177°C (320-350°F) and Zone 2 at 190-199°C (375-390°F). At steady state conditions, Zone 2 will be heated primarily by the resin exotherm. Normally, Zone 3 is unheated and allowed to cool by ambient conditions or it may be actively cooled. Line speeds up to 1.8-m/min (85-in/min) have been run with unidirectional profiles while maintaining good surface quality and low pull forces. It is feasible to run similar line speeds with mat reinforced profiles using the appropriate injection box design and well-guided reinforcements in combination with sufficient heater wattage and resin pump capacity.

**EHS Matters**

The two-component polyurethane resin systems described in this paper are compliant with the United States Environmental Protection Agency's Maximum Achievable Controls Technology (MACT) rules and do not use styrene or generate volatile organic compounds (VOCs). The engineering controls required to comply with local occupational safety and health guidelines for MDI-based isocyanate use are well-understood, easy to implement and have been widely used for decades in the automotive, construction and engineered lumber industries.

Joshi et al. (Ref. 5) were the first to report on EHS observations for polyurethane pultrusion. They measured locally elevated concentrations of MDI vapor at the exit of the pultrusion die. Based on this study, ventilation controls such as a canopy hood are recommended at the die exit and over all large volumes of mixed resin. In addition, atmospheric (or hygiene) monitoring should be done to ensure the ventilation controls are sufficient to keep local MDI concentrations below government-required safety limits.
Material Properties

In choosing a composite material for a specific application and, in turn, selecting the appropriate resin for the composite, the design engineer and end-user will focus initially on whether the resin and composite meet macroscopic material property requirements. Typically, the highest performing composite system will be chosen which meets processing goals and cost targets. Several previous studies (Ref. 3, 6-9) have reported on the excellent physical properties that can be attained with polyurethane resin based pultrusion profiles. However, these studies lacked direct comparison of PU to competitive resins. In an effort to more thoroughly benchmark PU performance, an extensive study was undertaken to pultrude vinyl ester (VE), unsaturated polyester (UPE), unsaturated polyester-urethane hybrid (UPE-PU) and polyurethane (PU) resins with identical reinforcement schedules.

Tensile Properties

The tensile properties of test profiles pultruded with VE, UPE, UPE-PU hybrid and RIMLINE SK 97007 polyol/SUPRASEC® 9700 isocyanate-based polyurethane (PU) resins are compared in Figure 5. The fiberglass lay-up is identical for all resins and uses standard multi-resin compatible sizing. The profile is 2.54-mm (0.100-inch) thick and contains two 300-g/m² (1.0-oz/ft²) continuous strand mats (CSM) and rovings for a total glass content of ~68 wt %. The tensile modulus and tensile strength of a composite in the fiber direction are dominated by the reinforcement and might be expected to be independent of resin type. However, the PU profiles exhibit clearly superior tensile modulus and strength in both the longitudinal and transverse directions compared to the other resin systems. The explanation of this significant property improvement is multifaceted. Polyurethane resins typically exhibit excellent wetting efficiency and adhesion to the glass reinforcement. Combining these effects with a tough resin, a nearly optimal synergy of stiffness and toughness between the PU resin and the glass reinforcement is created. As will be discussed later, the tough, stiff nature of PU composites can bring less obvious benefits as well in composite design latitude and in secondary operations.

Impact Performance

This synergy of stiffness and toughness is also observed in an evaluation of composite damage tolerance. In Figure 6, Dynatup instrumented impact curves are compared for identical composites of each resin system. As with the tensile properties, the PU composite displays superior toughness relative to the VE, UPE and UPE-urethane hybrid resin profiles. In this type of experiment, the area under the curve indicates the total energy absorption during dart penetration and the area up to the maximum load reveals the impact energy absorbed before catastrophic failure. The total energy absorption for PU profile was found to be 31 %, 39 % and 20 % higher than the VE, UPE and hybrid resin profiles, respectively (see TABLE 1). The more critical energy-to-maximum load was found to be 36 %, 118 % and 32 % higher for PU profiles compare to VE, UPE and hybrid profiles, respectively. For uses requiring both high stiffness and toughness, PU composites have a clear advantage to the end user.

Cost Analysis

Polyurethane resins have been or are being commercialized in a wide range of applications. Because of the outstanding performance of PU profiles, it is possible to re-engineer the reinforcements used to fabricate lighter weight and potentially less-expensive composites with PU compared to commonly used UPE. While the cost of PU resin is typically higher than UPE resin, the toughness, damage tolerance and strength of polyurethane resins bring the potential to simplify the lay-up and reduce the reinforcement cost in pultrusion profiles by replacing mat with rovings. Further, converting mat to rovings in the design will increase profile stiffness and liberate the potential to reduce the overall composite geometry. These two design elements together can actually offer a cost reduction for PU versus UPE. Additionally, the strength and toughness of PU pultrusions could translate into easier component assembly, reduced scrap, reduced warrantee costs and greater customer satisfaction.

To illustrate this benefit, an estimate is depicted in Figure 7 of the cost and weight savings at equivalent longitudinal stiffness for a pultruded I-beam that could be achieved by modifying the fiberglass construction. Such a beam 3.3-mm (0.130-inch) thick may contain three 300-g/m² (1.0-oz/ft²) continuous
strand mats (CSM) and have a total glass content of 59 wt %. Under the constant thickness assumption in this model, it is not possible to attain cost equivalence between PU and UPE profiles even when using only two 230-g/m² (0.75-oz/ft²) mats and filling the remaining profile space with rovings. However, a significant increase in the bending stiffness (+22%) of the beam will be obtained due to the extra rovings. On the other hand, both weight (-13%) and cost savings (-7%) may be achieved at equivalent beam stiffness by reducing the number and weight of mats AND the wall thickness (2.76-mm, 0.109-inch). Depending on design intent, structural components may be fabricated to take advantage of either the higher stiffness at constant cross-section or a reduced wall thickness with lower weight and reduced cost. This design latitude enables product improvement or differentiation at lower cost in materials without sacrifice of productivity.

**Environmental Exposure**
Verifying that a particular material meets the physical property requirements for an application is only one step in ensuring that material is fit for the purpose. Long term exposure to moisture, chemicals, ozone, electrical fields, cyclic or creep loads, UV or other radiation and temperature extremes are 'real world' hazards that must be considered before choosing a material for a particular use. Because they tend to perform well under such conditions, composites are used in many applications in which they are exposed to aggressive, potentially degrading environments. Therefore, evaluation of pultruded profiles in such environments is critical to ensure functionality is maintained over time.

**Water Absorption**
Many long term performance measures are linked to a material's propensity to absorb moisture, especially for those materials that are sensitive to hydrolysis. High water absorption can result in significant swelling stresses and subsequent fiber-matrix debonding, resulting in loss of composite strength and stiffness and providing opportunity for more water ingress and hydrolytic polymer degradation. As a first step in a larger program to study environmental effects, water absorption was measured for the PU, VE, UPE and UPE-PU hybrid composites of identical reinforcement lay-up described in Material Properties section of this paper (see Figure 8). The PU profiles exhibited substantially lower water absorption than the competitive composites – 42 %, 53 % and 36 % lower, respectively, than the VE, UPE and UPE-PU hybrid profiles. As described earlier, PU resins tend to wet fiberglass reinforcements well; hence, the void content of pultruded PU composites is typically very low (less than 1 %). In addition, the interfacial fiber-matrix bond strength of PU to fiberglass – with appropriately matched sizing – has been reported by Agrawal and Drzal (Ref. 11) to be quite high. These factors – low void content and high interfacial strength – are key drivers affecting water absorption and subsequent long term behavior.

**Sea Water Exposure**
In applications such as grating for off-shore oil platforms, sheet pile for sea walls and structural components for marine docks, profiles are obviously exposed to sea water. Compared to fresh or distilled water, sea water tends to be a more aggressive environment since the metal salts tend to catalytically promote hydrolysis. A preliminary study of exposure of VE, UPE, hybrid and PU profiles to artificial sea water is shown in Figure 9. Many factors could be chosen to judge long term performance. Since these exposure tests were limited to a three month time frame, a sensitive material characteristic was needed to differentiate the various composites. This study focused on the change of transverse flexural strength after exposure as this factor is strongly dependent on resin type. Not only do PU profiles display higher initial strength than the competitive resins, but they retain more strength over time than the VE, UPE and hybrid profiles. These general-purpose competitive resins all contain ester linkages in the polymer backbone which can hydrolyze over time, decreasing resin strength. The RIMLINE SK97007/SUPRASEC 9700 polyurethane resin used in this study does not contain ester chain linkages. In addition to hydrolysis, the salt water can diffuse into voids in the composites potentially causing resin-fiber delamination and additional strength and stiffness reduction. Longer term sea water exposure as well as more aggressive acid and base exposure tests are on-going.
UV Exposure

Many pultrusion applications involve outdoor use with potential for long-term exposure to ultra-violet (UV) light. As with hydrolysis, UV radiation can cause polymer breakdown and reduction in strength. In addition, UV degradation leads to discoloration which is usually assumed to indicate property reduction. VE, UPE, hybrid and PU profiles with identical reinforcement (2.54 mm thick with two layers of 230-g/m² CSM, ~72 wt % fiberglass) were exposed to UV and humid environments in a QUV chamber for 1000 hours. None of the resins contained UV stabilizing additives. The change in transverse strength is shown in Figure 10 for each type of composite. Although there is some scatter in the data, it is clear that the PU and hybrid resins exhibit better strength retention (10-15 % loss) versus the UPE (15-20 %) and the VE (25-30 %). Interestingly, the VE composite displays the smallest color change while the PU and hybrid UPE-PU exhibit the greatest yellowing, indicating that color shift is not necessarily a telling factor in strength degradation.

Many composites are ‘over-engineered’ to account for property loss over time and to maintain a sufficient ‘safety factor’ for end-use. Pultruded PU composites possess both higher initial strength and lower property loss with environmental exposure than the competitive VE, UPE and UPE-PU hybrid profiles. These observations reveal a significant opportunity for design engineers and pultruders to re-engineer their parts to take advantage of PU composite performance, producing a combination of lighter weight, more cost effective or stiffer components. These initial results give promise that PU composites will perform well in wet environment and outdoor, UV exposed applications and provide additional product differentiation to the pultruder and end-user. With judicious design, it is feasible to produce PU profiles that are not only tougher, stronger and lighter than competitive composites, but will remain so over time.

Secondary Operations

As with environmental exposure, performance of a component in ‘real world’ tests of cutting, machining, drilling, bonding and mechanical assembly is important to prove viability in an application. Polyurethane pultrusion technology is under development or has been commercialized in applications ranging from structural channels to tubes and beams to electrical components to window lineals and stiffeners to sporting goods. In each case, the pultruded profiles undergo some form of secondary operations before end use. For processes such as cutting and assembly, the toughness and durability of polyurethane composites can pay significant dividends in reduced scrap, ease of assembly, decreased labor or reduced assembly cost.

While studies of bearing strength and adhesive bonding are on-going, the results of an initial study into screw pull-out force are displayed in Figure 11. As with mechanical properties and damage tolerance, PU profiles exhibited significantly higher performance than the competitive resins. Using a modification of ASTM C1037, the force to remove a #10 self-tapping screw was measured. For profiles of identical fiberglass construction, the maximum pull-out force for PU profiles was found to be 31 % higher than for a hybrid UPE-PU composite and 43% and 79% higher than VE and UPE composites, respectively. When considering the force to initial crack formation, the difference is even more pronounced. The resistance to initial cracking of the PU composite was measured to be 99 %, 171 % and 48 % higher, respectively, than the VE, UPE and UPE-PU hybrid profiles. Once an initial crack is formed, a screw can easily work free over time with repeated loading. With such pronounced differences between PU profiles and the competitive materials, an opportunity may exist to rework attachment methods and lower assembly costs, perhaps eliminating plastic, wood or aluminium backing plates that are common in profile assembly. Additional benefits of using PU profiles which are mechanically assembled could include reduced labor costs, reduced warranty replacement and repairs, lower assembly scrap and improved product quality.
Conclusions
Polyurethane based composites have become an accepted alternative in the pultrusion industry, especially for high performance applications. PU pultruded profiles have proven to exhibit superior strength and toughness compared to VE, UPE and hybrid UPE-PU resins. PU profiles have also been found to exhibit promising preliminary results in environmental exposure tests and evaluation of assembly methods. These characteristics provide the design engineer, pultruder and end-user with broad latitude in PU composite design, process and fabrication at competitive costs, opening new opportunities in new markets – perhaps in some where composites have not been cost or performance competitive before the development of PU pultrusion resins. The modified pultrusion processing methods outlined in this paper will help most pultruders take full advantage of the performance of polyurethane resins. Further dissemination of these methods across the industry will help realize the full potential of the polyurethane resins to expand applications and broaden pultrusion into new markets.

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References
Figure 1 – Schematic of a Typical Polyurethane Pultrusion Set-up

Component Tank
Solvent Tank
Pneumatic Drive Cylinder
Dual-Action Variable Ratio Pump
Mixhead
Mix Tubes
Control

Figure 2 – Typical Two-Component Variable Ratio Metering Unit for Polyurethane Pultrusion
**Figure 3** – Variable Ratio Metering Pump and Mixhead for Polyurethane Pultrusion

- **Component Pressure Gauge**
- **Mixhead**
- **Component Mix Tube**
- **Good Mix**
- **Adjust Pump Ratio**

**ISO**

**POLY**

**Cool Entrance (4”, 10 cm)**
- <120 °F (<49 °C)

**Thermal Insulation**
- (reduce temp. gradients)

**Cool Exit**
- <250 °F (<120 °C)

**Zone 1**
- 320-350 °F
- (160-177 °C)

**Zone 2**
- 375-390 °F
- (190-199 °C)

**Cu Paste**
- (improve thermal conductivity)

**Figure 4** – Typical Die Set-Up for Polyurethane Pultrusion (0.9 to 1.0 meter long die)
Figure 5 – Tensile Properties of Vinyl Ester, Unsaturated Polyester, Unsaturated Polyester-Urethane Hybrid and Polyurethane Pultruded Profiles (ASTM D638, 2.54 mm thick, 2 x 300 g/m² continuous strand mat, ~68 wt % fiberglass)

Figure 6 – Dynatup Instrumented Impact Behavior of Vinyl Ester, Unsaturated Polyester, Unsaturated Polyester-Urethane Hybrid and Polyurethane Pultruded Profiles (average of 4 specimens, 2.54 mm thick, 2 x 300 g/m² continuous strand mat, ~68 wt % fiberglass)
### TABLE 1 – Instrumented Impact Results
(2.54 mm thick, 2 x 300 g/m² continuous strand mat, ~68 wt % fiberglass)

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**Figure 7** – Estimation of Cost, Weight and Stiffness of Unsaturated Polyester (UPE) and Polyurethane (PU) I-Beams with Various Fiberglass Constructions
**Figure 8** – Water Absorption of Vinyl Ester, Unsaturated Polyester, Unsaturated Polyester-Urethane Hybrid and Polyurethane Pultruded Profiles (2.54 mm thick, 2 x 300 g/m² continuous strand mat, ~68 wt % fiberglass)

**Figure 9** – Effect of Artificial Sea Water Exposure on Transverse Flexural Strength of Vinyl Ester, Unsaturated Polyester, Unsaturated Polyester-Urethane Hybrid and Polyurethane Pultruded Profiles (2.54 mm thick, 2 x 300 g/m² continuous strand mat, ~68 wt % fiberglass)
**Figure 10** – Effect of QUV Exposure on Transverse Flexural Strength of Vinyl Ester, Unsaturated Polyester, Unsaturated Polyester-Urethane Hybrid and Polyurethane Pultruded Profiles (2.54 mm thick, 2 x 230 g/m² continuous strand mat, ~72 wt % fiberglass)

**Figure 11** – Screw Pull-Out Force for Vinyl Ester, Unsaturated Polyester, Unsaturated Polyester-Urethane Hybrid and Polyurethane Pultruded Profiles (modified ASTM D1037, #10 self-tapping screw, 2.54 mm thick, 2 x 300 g/m² continuous strand mat, ~68 wt % fiberglass)
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