

KEY FACTORS OF THE PEEL PLY SURFACE PREPARATION PROCESS

David Klapprott, Helen Li, Raymond Wong and George Geisendorfer Henkel Corporation Bay Point, California 94565 USA

ABSTRACT

Nylon or polyester fabrics are used as release plies to create, upon removal, activated surfaces optimized for bonding of composite structures. However, this method of using peel ply to create a bondable surface lacks reliability. To improve on the consistency issue, the prevalent practice is to add a sanding or grit blasting step to ensure satisfactory surface preparation for bonding. The industry's ultimate objective is to develop a surface preparation material and process not requiring any additional surface preparation. The research effort of this paper is focused on identifying those peel ply material and processing characteristics that control the quality of the released surface. The complete research results are discussed in this paper.

Additionally, this paper will also discuss the development of an alternative peel ply, Hysol[®] EA 9895, which greatly improved the consistency of the release substrates and eliminated the need for secondary surface preparation steps.

KEY WORDS: Adhesives/Adhesive Bonding, Resins/Materials – Epoxy, Surface Preparation Materials/Processes

1. INTRODUCTION

There are two primary methods for adhesive bond preparation of the surface of a pre-cured carbon fiber composite; surface abrasion and the use of release ply fabric; i.e., peel ply. Of the two, peel ply is the most commonly used, not only saving significant amounts of labor but also creating a more evenly treated surface than hand or machine abrasion. However, general industry experience has shown that this process lacks reliability with respect to the performance of the resulting adhesive bond.

Hysol[®] Turco[®] Henkel Corporation Aerospace Group P. O. Box 312 2850 Willow Pass Road Bay Point, CA 94565 USA Phone 925.458.8000 Fax 925.458.8030

www.henkel.us www.aerospace.henkel.com



The lack of reliability has been investigated widely with the majority of results pointing to surface contamination of the peel ply fabric. For instance, Hart-Smith et al (1) found adhesive property reduction was due to silicone contamination of the fabric. The contaminant source was probably the lubricants applied to the fibers in the weaving of the peel ply fabric. Silicone contaminants have also been detected on fabrics screened in Henkel's laboratories.

However, Henkel found significant variation in adhesive performance on bonding surfaces generated using contaminant free peel ply fabrics. The level of this variation was related to both the type of fabric used and to the particulars of the composite curing process. Research was conducted to understand the reasons for this variation and, hopefully, to develop a more predictable peel ply process.

"Copyright 2004 by Henkel Corporation. Published by Society for the Advancement of Material and Process Engineering with permission".)

2. PROCESS MODEL

The hypothesis used in this work is that the adhesive performance variation was not due to the variable level of adhesion of resin to the peel ply fabric but rather the degree of impregnation of the fabric and the crack toughness of the cured resin. Wetting and adhesion of a surface by a liquid requires that the liquid's surface tension (γ) be less than the surface's critical energy (γ_c). Typical γ values for epoxy are in the 4.7 x 10⁻² to 5.5 x 10⁻² N/m (47 – 55 dynes/cm) range while γ_c for a polyester fiber lies in the 4.2 x 10⁻² to 4.3 x 10⁻² N/m area. The γ_c range for nylon fibers is 4.6 x 10⁻² N/m range.

Rather than a variable level of adhesion controlling the performance level, we hypothesized that this variation correlated with a variable level of peel ply fiber left on the bond surface after fabric removal. That fibers remain on the surface can be easily seen using SEM analysis. Figure 1 illustrates this for a bond surface generated using a dry polyester fabric on a toughened epoxy matrix prepreg system.

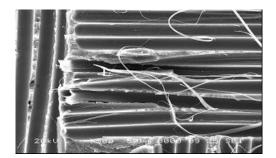


Figure 1: Residual Polyester Fiber on Bonding Surface



A processing model (Figure 2) was hypothesized which might account for the variation in the level of fiber remaining on the bonding surface.

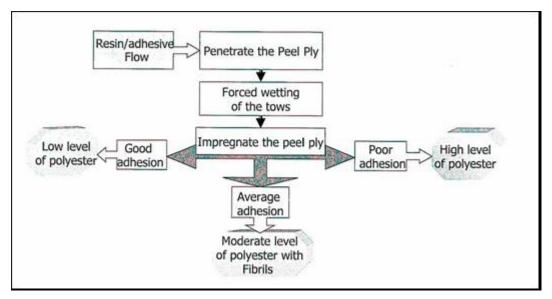


Figure 2: Process Model

The amount of residual fiber, according to the model, depends upon the degree of resin encapsulation of the fabric. The level of fabric encapsulation is dependant upon both fabric and process characteristics. Variation in the weave density, filament count in the tow and filament diameter could cause variation in the degree of encapsulation. Likewise, variation in the cure viscosity profile caused by both the inherent cure kinetics of the resin system and the temperature ramp profile of the particular cure can produce different levels of fabric encapsulation.

The encapsulation process can be envisioned as follows: as the temperature of the part increases, resin flows into the dry peel ply fabric. This flow pattern has two sequential parts: the initial flow through (normal to) the fabric filling the interstitial volumes between the fiber tow bundles and a sequential "back" flow into (radial to) the bundle itself. The influence of the fabric weave density can be seen in Figure 3.



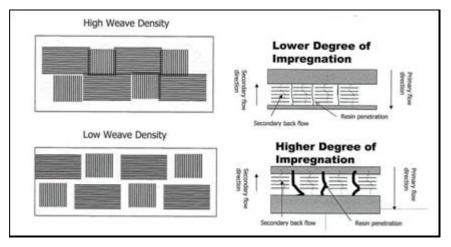


Figure 3: Effect of Weave Density on Resin Flow

The higher weave density (yarns/inch) fabrics have more but smaller interstices per unit area compared the lower weave density fabrics. Subsequently, the amount of initial flow is reduced causing a tendency towards lower amounts of resin encapsulation of the polyester fibers by the resin.

The back flow through the individual tow bundles is dependent upon the diameter of the individual fibers themselves and to the number of fibers per tow. As the diameter decreases and the number of fibers per tow increases, the time required to completely encapsulate the bundle increases. Fabrics made with small diameter fibers and high tow count per inch tend to cause incomplete filling of the peel ply fabric as depicted in Figure 4.

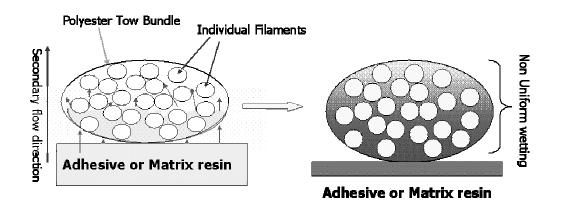


Figure 4: Resin Flow into a Tow Made up of Small Diameter Fibers



Those fabrics with large diameter fibers and a lower tow count have less resistance to back flow. This results in higher levels of filling of the fabric construction as depicted in Figure 5.

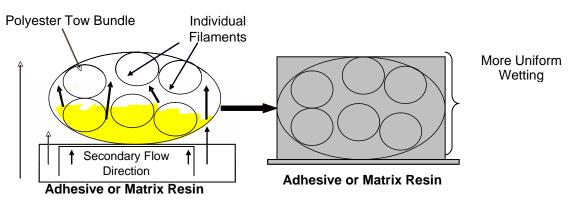


Figure 5: Resin Flow into a Tow Made up of Large Diameter Fibers

The amount of back flow is also dependent upon the cure kinetics of the resin system. As the resin cures, its viscosity increases as does the time needed to completely encapsulate the fiber bundle. Those systems that are very reactive or cure schedules that tend to keep the viscosity of the resin system high throughout the cure tend to decrease the level of fabric encapsulation.

When peel ply layers with high levels of resin encapsulation are removed from the cured composite surface, there is a strong tendency for fracture to occur in the resin system, away from the fiber bundles themselves. However, with incomplete fabric encapsulation, weakened areas occur within the fiber bundle itself. Fracture tends to occur within the weak areas leaving variable amounts of peel ply fiber (Figure 6). Since the adhesion of the adhesive resin to the peel ply fibers is poor, insufficient bond properties are generated when the adhesive bond is formed.

The final variable that appears to be important is the fracture energetics of the encapsulating resin itself. If this is high, the fracture plane may be driven into the fiber bundle, also generating residual peel ply fibers on the bonding surface.

3. EXPERIMENTAL

Eight nylon peel ply fabrics and ten polyester fabrics were screened for performance in this effort. These were representative of the products of suppliers in both Europe and the U.S. Preliminary screening consisted of measuring the honeycomb peel strength using CF composite skins bonded to adhesive stabilized Nomex[®] honeycomb core. The specifics of the construction are:



Adhesive: EA 9695, 0.05K Prepreg: Cytec HTA/977-2, 2X2 Twill, 285 g/m² Composite Skins: 3 plies, [+45, 0,-45], pre-cured with peel ply on bonding surface removed Honeycomb Core: Nomex[®] HRH-10, 96 kg/m2, 4.76 mm cell size, 15.9 mm thick

4. RESULTS

4.1 Dry Peel Ply Very poor peel strengths were measured with all of the nylon peel ply fabrics screened. Surface analysis for contaminants indicated the presence of silicone on the primary nylon fabric to be screened. Consequently, the work was focused on polyester fabrics, which had not shown any surface contamination.

Six polyester fabrics formed the central portion of the investigation. The construction characteristics of these fabrics are shown in Table 1:

	Tow Count		Fibers/Tow		Fiber Diameter, µ		Weight
Fabric	Warp	Fill	Warp	Fill	Warp	Fill	g/m ²
A	70	50	50	50	19	19	88.0
В	76	54	32	35	23	23	97.7
С	103	87	30	35	16	16	65.5
D	60	38	50	50	23	23	111.4
E	120	64	32	34	16	22	83.1
F	54	42	55	55	23	23	112.4

Table 1: Dry Polyester Peel Ply Construction

Comparison of the peel ply fabric surface after removal from the cured composite illustrates the impact of fabric density (Figure 7). The fiber bundles in the stripped moderate density fabric are shredded indicating that a significant amount of fiber was left on the bonding surface. The fiber bundles in the low density fabric are more whole indicating that little fiber is left on the bonding surface.

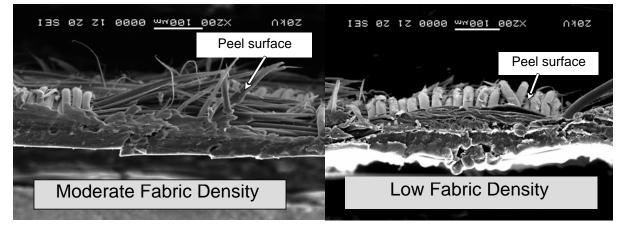
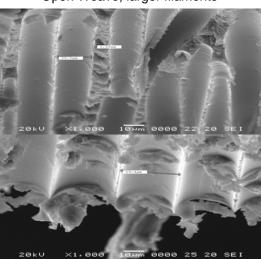


Figure 7: Bond Surfaces Generated High and Low Density fabrics



The net impact on the bonding surface can be seen in Figure 8. The low density fabric leaves a cleaner surface with little retained peel ply fibers. Considerable fiber remains on the bonding surface after removal of the tighter weave, smaller filament fabric.



Open Weave, larger filaments

Tight Weave, small filaments

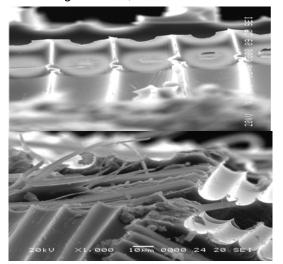


Figure 8: Bond Surfaces Generated High and Low Density fabrics

The density characteristics of the fabrics were characterized by calculating their unoccupied volume. This was calculated by measuring the thickness of the fabric and factoring in the tow count, fiber count per tow and the fiber diameter. The unoccupied volume of the six polyester fabrics roughly correlates to measured honeycomb peel strength (Figure 9).

Re-examining the model, it can be seen that the fracture locus depends not only upon the degree of filling of the peel ply fabric but also upon the fracture resistance of the resin that fills the fabric. A high toughness resin system would tend to drive the fracture location into the peel ply fabric even though it was completely filled. Recent improvements in the fracture toughness of composite resin systems tend to make the generation of fiber free surfaces more difficult compared to the first generation of composite resin systems. Control of the fracture location by regulating the fracture toughness of the resin layer immediately next to the peel ply surface is the next area of improvement.



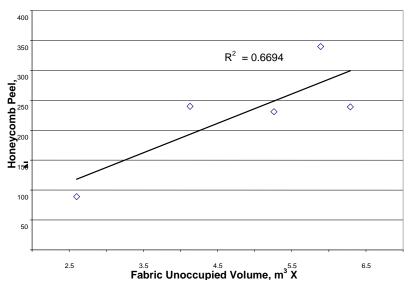
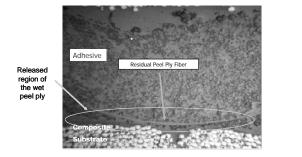


Figure 9: Influence of Fabric Unoccupied Volume on Honeycomb Peel

4.2 Pre-impregnated Peel Ply Systems, Wet Peel Ply The objective of this effort was to develop a system that would eliminate residual peel ply fibers on the bonding surface by controlling the location of the fracture during peel ply removal. An additional objective was to develop a system that was easy to remove from the composite surface eliminating the composite fiber damage that has been associated with the use of some of the dry peel ply fabrics. Of course, retention of good bond properties, such as G_{Ic}, was critical to the success of this project.

This objective was met by the development of an impregnating resin used in Hysol[®] EA 9895 with reduced crack toughness compared to both the current generation of tough prepreg resins and toughened adhesive systems. The reduced resin toughness directs the failure locus, during peel ply removal, to the region between the peel ply and the outer layer of carbon fibers. The peel ply is removed without leaving residual fibers on the composite surface. This can be seen by comparing cross-sections of bonds made with this new resin system and EA 9895, a toughened adhesive resin system (Figure 10).





Released region of the wet peel ply



a) Toughened Adhesive Resin

b) EA 9895 Resin

The lowered resin toughness of the impregnated peel ply does not negatively impact the toughness of the subsequent adhesive bond. This can be seen by comparing both the G_{lc} and G_{llc} properties of bonds made with various peel ply combinations (Figure 11).

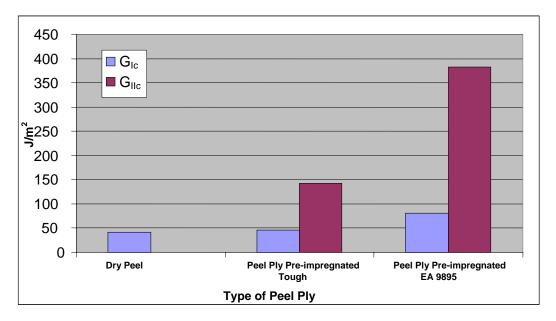


Figure 11: Effect of Optimized Peel Ply on G_{lc} and G_{llc} Performance



The G_{lc} performance of bonds made with either dry peel ply or peel ply pre-impregnated with a tough resin system is considerably lower that that of the bond made using the peel ply pre-impregnated with EA 9895 resin system. Values for the specimens made using the EA 9895 system are roughly double those made using dry peel ply. G_{llc} performance is similarly increased using the EA 9895 peel ply system with performance levels three times that of bonds made using a tough resin impregnated peel ply. The choice of resin system also influenced the bond fracture location. Bonds made using a dry peel ply prepared surface failed at or very near the adhesive/composite interface. In contrast, bonds made using the EA 9895 wet peel ply system failed cohesively within the adhesive layer itself (Figure 12).

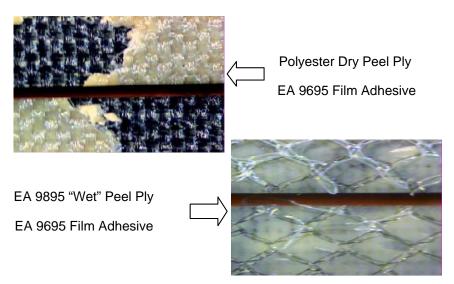
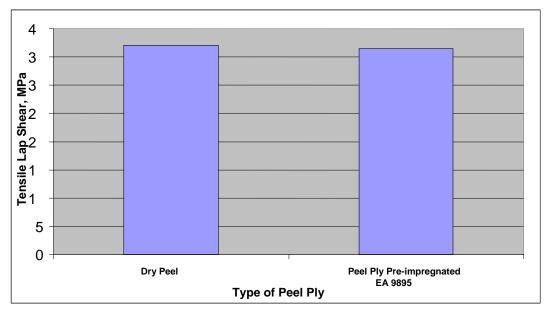
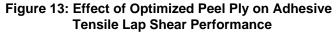


Figure 12: G_{Ic} Fracture Surfaces

It is important to note that the lowered resin toughness of the impregnated peel ply does not negatively impact the strength of the subsequent adhesive bond. This can be seen by comparing both the Tensile Lap Shear properties of bonds made with various peel ply combinations (Figure 13).







5. CONCLUSIONS

The work performed to date suggests that the performance and failure location of composite bonds made with peel ply prepared surfaces is strongly dependent upon the construction of the fabric, the ability of the impregnating resin to fully impregnate the fabric and the toughness of the impregnating resin system. Non-optimum combinations of these variables causes residual peel ply fiber on the bonding surface and fracture at or very near the adhesive/composite interface. Poor adhesion and the concomitant lowered levels of bond performance are the result. Conversely, optimum combinations of these variables lead to more activated bonding surfaces and, therefore, significant performance and failure mode improvements.

The optimum performance was produced when a high porosity fabric, comprised of a low count of tows consisting of large diameter fibers, was pre-impregnated with a more brittle resin system. The result of this optimization is Hysol[®] EA 9895. When used, all of the peel ply fiber was removed when the peel ply was stripped from the surface. The resultant bonds had fracture toughness levels from 2



to 3 times that of bonds made with either a dry fabric or fabric pre-impregnated with a tough resin. Use of this optimized system was associated with a change in fracture mode from an apparent interfacial case to cohesive within the adhesive.

6. REFERENCES

1) L.J. Hart-Smith, G. Redmond, and M.J. Davis, The Curse of the Nylon Peel Ply, <u>41st International</u> <u>SAMPE Symposium</u>, March 24-28, 1996, pp. 303-317