

Quantitative Assessment of Reactive and Non-Reactive Binder Systems on First Ply Adhesion for Dry Fiber Placement Processes

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Abstract. Dry fibers or fabrics do not possess an inherent tack, when compared to prepreg materials. In order to fixate dry fiber fabrics onto tooling geometries, auxiliary binder systems are necessary. These tackifying agents vary in terms of chemical composition and reactivity, processing parameters and appearance. One key aspect for most automated fiber placement technologies (incl. Dry Fiber Placement; Abr. DFP) is the adhesion of reinforcement materials on tooling surfaces, also referred to as first ply adhesion. Insufficient prepreg or binder adhesion will lead to fiber slippage thus increased scrap rates and is therefore crucial for high class composite performance. This study reveals quantitative insights on binder performance and indicates that the treatment with solvent-based release agents reduces first ply adhesion by up to 78%. Furthermore, it shows, that elevated tooling temperatures reduces binder adhesion by up to 49%.

Introduction.

When producing structures out of fiber reinforced polymers (FRP), textile fabrics manufactured, distributed and stored as rolled goods are often used. During cutting of those textile fabrics, significant quantities of trimming waste occur. This waste material does not only increase the end products cost, it also increases its ecological footprint. Technologies such as the Automated Tape Laying (ATL) [1], the Automated Fiber Placement (AFP) [2] or the Advanced Ply Placement (APP) [3] reveal a high potential for more material- and therefore cost-efficient composite production (see Figure 1). In order to reduce trimming waste, these robotized manufacturing technologies are using different pre-cut reinforcement materials with discrete material widths (e.g. AFP: 6.35 mm; ATL / APP: 300 mm) [4]. When looking at AFP layup, in general unidirectional tapes are used and distinguished by means of their matrix material or applied tackifying agent. While pre-impregnated thermoset tapes are the most commonly used fiber reinforcement type for AFP processes, thermoplastic tape materials or dry fabrics are gaining increasing interest within industrial applications [5].

Especially, preform manufacturing using Dry Fiber Placement (DFP) is an emerging technology, which enables high mechanical and cost-competitive performance for composite components. During DFP layup, continuous unidirectional, pre-bindered fabrics are placed directly onto a tooling surface in a robot-assisted layup process, in order to create a near-net-shape preform. With this approach highly functionalized preforms including a load-oriented fiber layup, reduced fiber crimp and undulations can be created [6]. By using DFP technologies additional cost of prepregging, refrigerated storage and autoclave processing can be significantly reduced. Furthermore, production of large-scale structures is facilitated, due to the missing out-time limitations of classical prepreg materials [7]. In comparison to pre-impregnated materials, dry fabrics need to be coated with an auxiliary tackifying agent, in order to prevent displacement during automated layup. This is due to the fact, that dry fabrics do not possess an inherent tack [8].

Various authors have studied the different effects of tackifying agents on mechanical and rheological performance [9]. The tensile strength of composite components is dominated by the tensile properties of the reinforcing fibers. For that reason, studies on tensile composite properties in regards to the effects of binder system are rare. Geßler et al. [10] and Beier [11] have shown, that the tensile properties of a composite laminate are barely affected.

Henning [12] and Schulz et al. [13] have analyzed bending properties, such as bending strength and modulus, of pre-bindered composite laminates. It has been shown, that bending properties were reduced by up to 15 % for all of the binder systems tested. The interlaminar shear strength is a quality criterion, which describes the strength of adjacent layers within a composite part. Since tackifying agents are placed exactly within two adjacent layers, it can be assumed, that the binder system has a major effect on these properties. Studies of Shih and Lee [14], Hillermeier and Seferis [15], Brody et al. [16], Tanoglu et al. [17], Bulat and Heieck [18] and Henning [12], have all shown, that elevated binder fractions will lead to reduced interlaminar shear strength. As mentioned above, the utilization of dry preforms and subsequent application of Liquid Resin Infusion (LRI) methods, such as the Vacuum Assisted Resin Infusion (VARI) or the Resin Transfer Molding (RTM), offers great potential for cost savings and superior material flexibility. But beyond the mechanical performance losses, the utilization of tackifying agents also affects preform permeabilities. Poor impregnation characteristics and processing limitations of DFP preforms during Liquid Resin Infusion methods offer significant drawbacks and are focus of recent research [19,20].

As just shown, different mechanical effects of auxiliary binder systems have been thoroughly studied by various authors. However, the adhesive properties of auxiliary tackifying agents have a significant effect on the accuracy during fiber layup and therefore on preform quality. Especially, the adhesion of reinforcement materials on tooling surfaces, which is also referred to as first ply adhesion, has a major influence on the subsequent process stability. Insufficient prepreg or binder adhesion will lead to preform slippage and increased scrap rates. Due to missing information on first ply adhesion, these aspects will be quantitatively assessed and discussed within this paper.

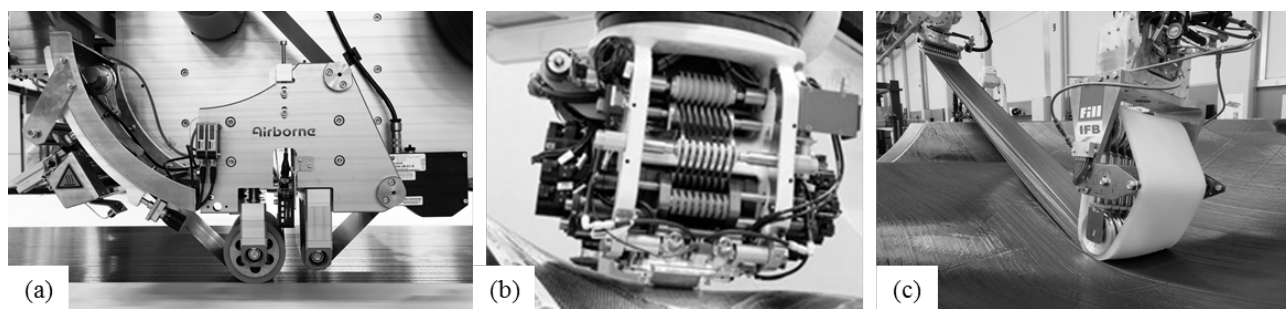


Figure 1: Automated Composite Manufacturing (a) ATL [21] (b) AFP [22] (c) APP [23]

Materials and Methods.

Materials. E-Glass fabric [24] was purchased from R&G Faserverbundwerkstoffe GmbH (Waldenbuch, Germany). It was a plain-woven fabric with an areal weight of 130 g/m² (50 wt% in warp and 50 wt% in weft direction) from 34 tex yarns made of Vetrotex EC 9 glass fibers with a density of 2,6 g/cm³. It was coated with a silane finish and had a fabric width of 25 mm (±1 mm). As reference material the unidirectional carbon fiber prepreg material C U600-0/SD-E501/33% with an areal weight of 600 g/m² [25, 26, 27] was purchased from SGL TECHNOLOGIES GmbH (Meitingen, Germany).

Within this study seven different reactive and non-reactive tackifying agents (three powder binders, two veil binder systems, one hot-melt binder and one spray tackifier) were analyzed concerning their tackifying behavior. The binders vary in terms of their physical appearance, activation temperature and chemical composition (see Table 1). EPIKOTE™ Resin 05311 [28] was supplied by HEXION (Esslingen, Germany). It was a white, solid epoxy resin powder based on Bisphenol-A with a grain size between 90 – 125 µm and an activation temperature of 102±5 °C. EPIKOTE™ Resin 05390 [29] was also supplied by HEXION (Esslingen, Germany). Just as EPIKOTE™ Resin 05311, it comes as a white epoxy-based powder, which is applied as hot-melt system in order to stabilize composite reinforcements. EPIKOTE™ Resin 05390 has a particle size between 50 – 90 µm and a softening point of 90±15 °C. Araldite® LT 3366 [30] has been purchased from Huntsman Advanced Materials (Switzerland) GmbH (Basel, Switzerland). It is a solid,

Bisphenol-A based, high molecular weight epoxy resin and comes as a white powder with a grain size between 160 – 200 μm . The activation temperature is stated as 190 $^{\circ}\text{C}$. SAERfix[®] EP [31] was purchased from SAERTEX GmbH & Co. KG (Saerbeck, Germany). It is a self-adhesive veil and shows sufficient tack at room temperature, so that binder activation is not necessary. The veil has an areal weight of 12 g/m^2 . EPIKOTE[™] Resin MGS PR685 [32] was supplied by HEXION (Esslingen, Germany) and is delivered as a high viscous resin component based on Bisphenol-A. Due to its high viscosity ($\text{RT} \gg 30\,000 \text{ mPa}\cdot\text{s}$), application at elevated temperatures with a pneumatic hot-melt glue gun (BÜHNEN HB700 K spray [33]) is necessary. Spunfab PA 1541 [34] was purchased from Hänsel Verbundtechnik GmbH (Iserlohn, Germany). PA 1541 is a thermoplastic copolyamide veil with a melting temperature between 87 – 100 $^{\circ}\text{C}$ and an areal weight of 12 g/m^2 . AIRTAC 2E [35] was purchased from Haufler Composites GmbH & Co. KG (Blaubeuren, Germany) and is a rubber-based spray adhesive for temporary fixation of composite materials. After evaporation of the solvents, it exhibits tackiness at room temperature without further activation.

Table 1: Summary of tackifying agents

Name	Abbreviation	Supplier	Bindertype	Chemical Composition	Appearance	Activation Temperature [$^{\circ}\text{C}$]
EPIKOTE 05311	E5311	HEXION	Reactive	Bisphenol A	Powder	97 - 107
EPIKOTE 05390	E5390	HEXION	Reactive	Bisphenol A	Powder	75 - 105
Araldite LT3366	LT3366	HUNTSMAN	Reactive	Bisphenol A	Powder	160 - 200
SAERfix EP	SAER	SAERTEX	Reactive	N.A.	Veil	RT
MGS PR685	PR685	HEXION	Reactive	Bisphenol A	Hot-Melt	RT
PA1541	PA1541	Spunfab	Non-Reactive	CoPolyamide	Veil	87 - 100
AIRTAC 2E	AIR2E	AIRTECH	Non-Reactive	Rubber-Based	Spray	RT

In order to assess and compare the tackifying behavior of the binder systems, three different substrate materials have been identified (Figure 2), which shall represent state of the art metallic and composite tooling materials. Aluminum (AlMg3, material reference: 3.3535, density: 2.66 g/cm^3) and stainless-steel sheets (V2A, material reference: 1.4301, density: 7,9 g/cm^3), were purchased from Rayonic Laserschneidtechnik GmbH (Leipzig, Germany). The aluminum sheets are made from an aluminum-magnesium alloy with a magnesium content of 3%, which are suitable for tool manufacturing. Carbon fiber reinforced sheets were purchased from R&G Faserverbundwerkstoffe GmbH (Waldenbuch, Germany). The sheets are manufactured with HT carbon fiber prepreg material in a hot-pressing process. Top layers contain 200 g/m^2 carbon fiber prepreg (twill weave) and one core-layer of unidirectional fabric.

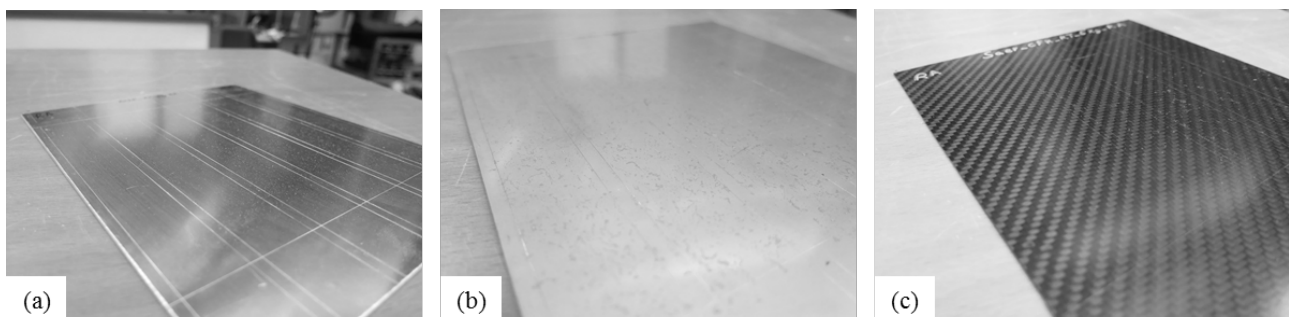


Figure 2: Substrate materials (a) AlMg3 (b) V2A (c) CFRP

Two different release agents and their effect on binder adhesion have been compared within this study. LOCTITE[®] FREKOTE 770-NC[™] [36] was purchased from Henkel AG & Co. KG (Düsseldorf, Germany). It is a clear, colorless solvent based release coating, suitable for epoxy and polyester resins, vinyl ester resins and thermoplastic materials. ZYVAX[®] 1070 W [37] and ZYVAX[®] FreshStart [38] was supplied by Chem-Trend Deutschland GmbH (Maisach Gerlinden, Germany).

ZYVAX® 1070 W is a silicone-free, water-based release agent specifically formulated to meet high performance aerospace requirements. It is suitable for use with all tooling types and molding processes. ZYVAX® FreshStart is a solvent-free mold and part cleaner for tooling preparation prior to application of ZYVAX® 1070 W.

Methods.

The different binder systems were manually applied on the E-Glass fabric. Due to industrial standards, a binder fraction of 5wt% [39] was aimed for and documented with a precision scale TGD 50-3C [40] from KERN & Sohn GmbH (Balingen, Germany) with a measuring accuracy of 0.001 g. Powder binders (E5311, E5390, LT3366) were applied using a metallic sieve for homogenous fabric coating, while veil binders (SAER, PA1541) were precut and manually placed onto the fabric. For manual application of the hot-melt (PR685) and spray tackifiers (AIR2E), the fabric samples were placed onto a horizontal surface and the adhesives were sprayed onto the specimen with an approximate distance of 300 mm. For thermal activation of E5311, E5390, LT3366 and PA1541 the test specimen were placed into a universal lab oven Memmert UF260plus (Schwabach, Germany) [41] with a setting accuracy temperature of ± 0.5 °C. The activation temperature was set to 200°C and activation time to 10 minutes. Preform consolidation was achieved by the placement of two different weights (50 N eq. 1.2012 kN/m²; 100 N eq. 2.4024 kN/m²) onto a metallic caul plate. Room temperature binders (SAER, PR685, AIR2E) do not need additional thermal activation. Equivalent consolidation forces of 50 N and 100 N were applied. The reference CF prepreg fabric was placed onto the tooling surface at room temperature and consolidated accordingly. Furthermore, the environmental conditions were monitored and documented throughout the entire study using VOLTcraft DL200T (Hirschau, Germany) [42]. In average the room temperature was at 25.7°C while the relative humidity was monitored at 49 %.

For quantitative measurement of peel forces a universal testing machine Inspekt table 20-1 [43] from Hegewald & Peschke Meß und Prüftechnik GmbH (Nossen, Germany) equipped with a 200 N loadcell 1-U9C/200N [44] from HBM Deutschland (Darmstadt, Germany) was used. The software LabMaster from Hegewald & Peschke Meß und Prüftechnik GmbH (Nossen, Germany) was used for data documentation and visualization with a measuring resolution of 50 Hz.

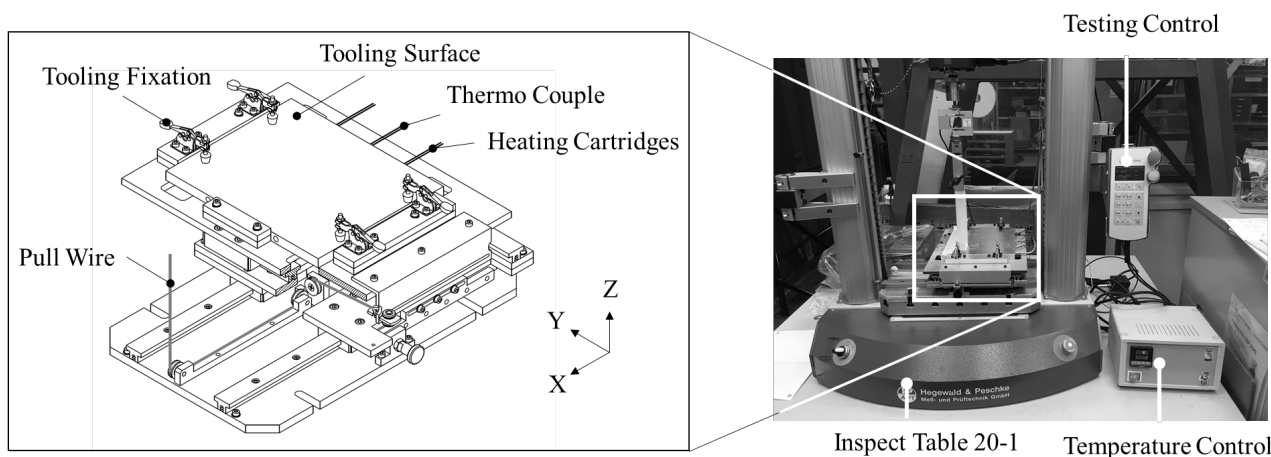


Figure 3: Test Rig acc. DIN 28510-1

In order to analyze and compare adhesive forces on tooling surfaces, a test set-up according to DIN EN 28510-1 [45] was chosen. This standard specifies a 90° peel test for the determination of the peel resistance of a bonded assembly of two adherends, in which at least one of the adherends is flexible. In order to achieve a constant peel angle of 90° a test rig has been developed, in which different tooling materials, surface temperatures and up to 6 specimens can be tested (see Figure 3 and Figure 4). In order to control the tooling temperature, three cylindrical heating cartridges [46] and a NiCr-Ni Type K thermocouple [47] were integrated into the test rig. Different substrate materials (AlMg3, V2A; CF composite tool) were clamped onto the test rig and tooling temperatures were set to room temperature (Abr. RT) at 25°C and elevated temperatures at 40°C (Abr. ET). For

each test series a minimum number of five samples was tested. Characteristic values, such as maximum and minimal peeling forces, average peel forces and the standard deviations have been documented (see Figure 5, Table 2, Table 3 and Table 4). Test specimen were cut to a length of 250 mm. The width of 25 mm was given by the chosen E-Fabric. According to DIN EN 28510-1 the traverse speed of the universal testing machine was set to 50 mm/min.

In total five benchmark measurements have been conducted within this study, taking into consideration the various binders, release agents, tooling materials, tooling temperatures and consolidation forces. Within the first test series, the influence of the release agent LOCTITE® FREKOTE 770-NC™ (Abr. F770NC) was compared to an untreated aluminum sheet (AlMg3; RT; 50 N). The second test series analyzes the influence of tooling temperatures on first ply adhesion on a pretreated aluminum tool (AlMg3; F770NC; 50 N). Within the third series two different consolidation forces were applied during sample manufacturing without prior application of a release agent (AlMg3, w/o RA; RT). In order to study the influence of different tooling surfaces on first ply adhesion only SAERfix EP and the reference prepreg material were considered for comparison. Three tooling surfaces have been tested with and without prior treatment with F770NC (RT; 50N). Ultimately, the water-based release agent ZYVAX® 1070 W was compared against LOCTITE® FREKOTE 770-NC™ and benchmarked against an untreated aluminum sheet (AlMg3; RT). Spunfab PA1541 was considered for this test, due to good adhesive behavior and common applicability in scientific and industrial applications.

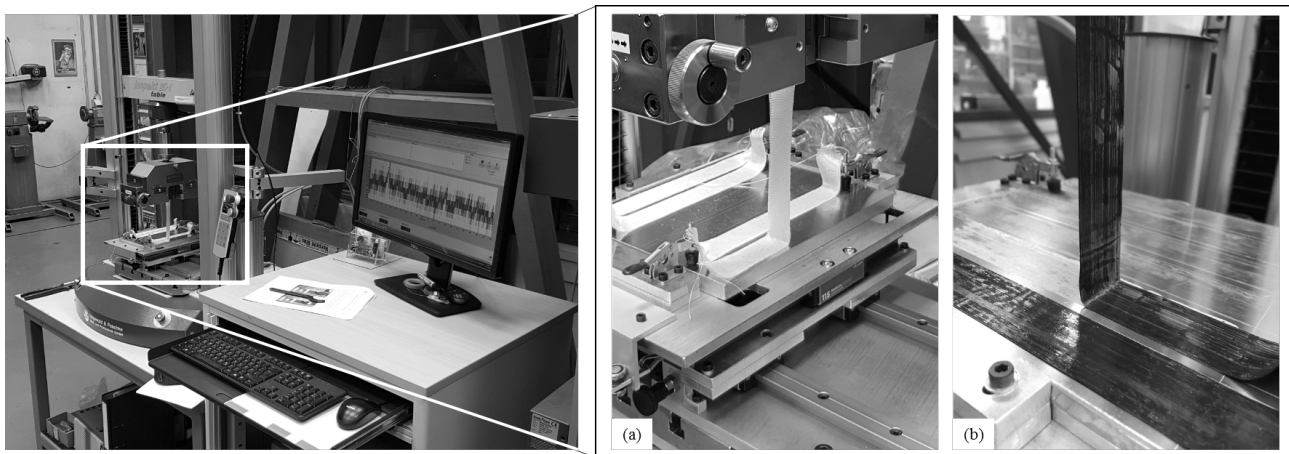


Figure 4: Test Set-Up (a) E-Glass Fabric (b) CF Prepreg

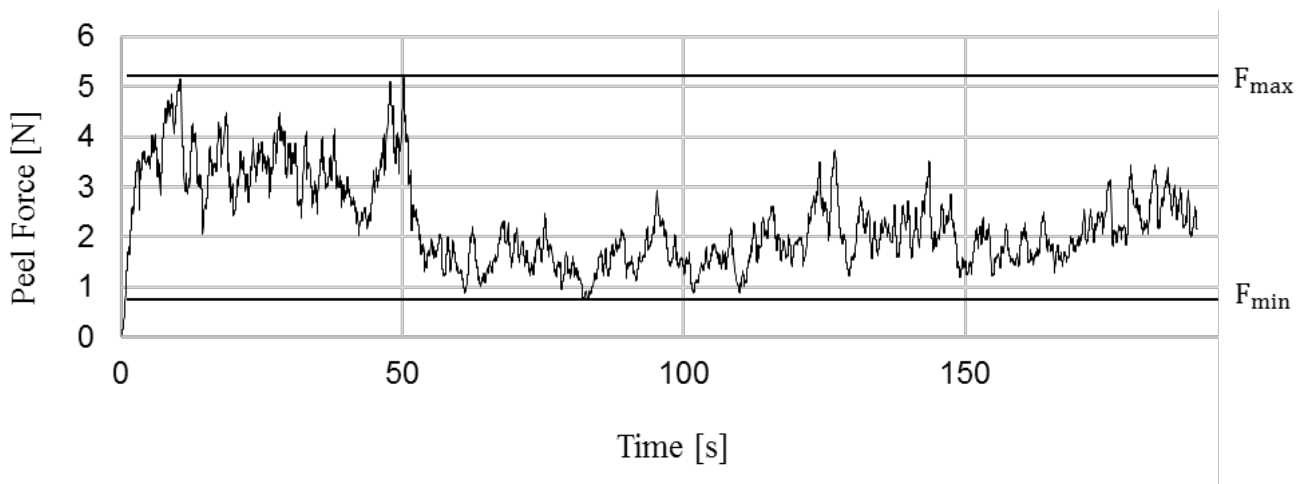


Figure 5: Force-Time Diagram acc. DIN 28510-1 for PA1541-AL-w/o_RA-50N

Results.

As it can be seen in Figure 6 (a) and Table 2, the solvent based release agent F770NC has a significant impact on first ply adhesion, when applied to aluminum tooling surfaces at room temperature testing conditions. In average the adhesive forces were reduced by 77.78%. When compared to each other, the tackifying agents LT3366 (-70.31%) and PR685 (-73.85%) showed smallest impact. Only SAER was able to increase its tack by +52.07%. Pretreatment with F770NC showed only little influence on the reference prepreg sample (-18.67%)

When analyzing the influence of elevated tooling temperatures on first ply adhesion on a pretreated AlMg3 tool, Figure 6 (b) and Table 1 shows, that for all tested specimen the tack was reduced. In average peel-off forces were reduced by 48.68%, whereas E5311 (-1.87%) and LT3366 (-40.76%) showed smallest reductions, while SAER (-73.19%), PR685 (-86.95%) as well as the reference sample (-69.68%) showed highest impact. For E5390 an insufficient number of valid samples has been measured, so it cannot be considered in the evaluation.

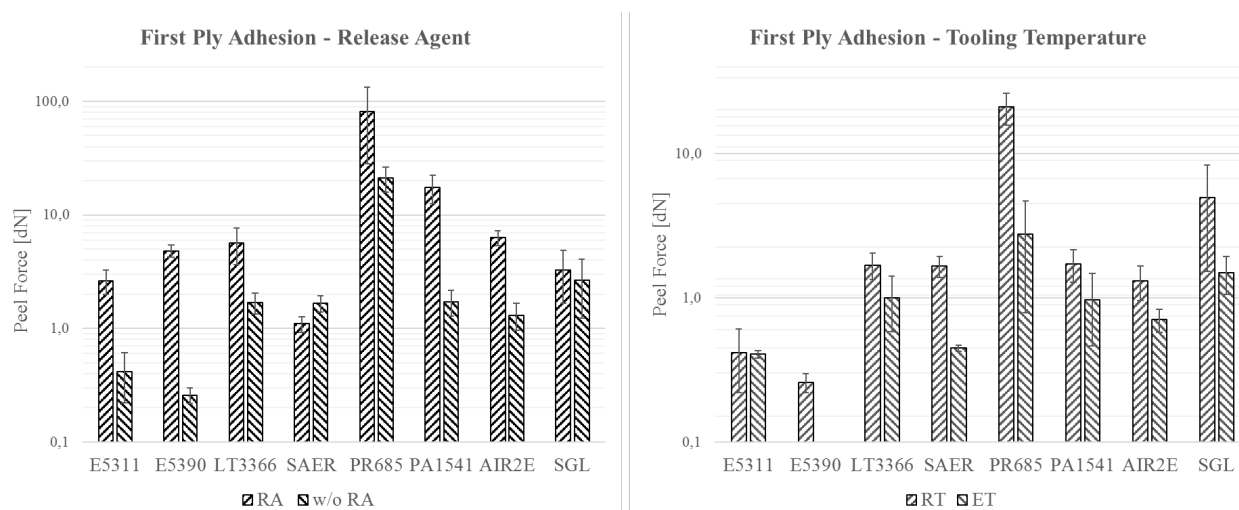


Figure 6: Influence on First Ply Adhesion (a) Release Agent (b) Tooling Temperature

Consolidation forces (50 N and 100 N) were analyzed in Figure 7 (a) and Table 2. Samples were applied on an untreated AlMg3 tool and tests have been conducted at room temperature conditions. The quantitative measurements showed inconsistent results. While adhesion was reduced for E5311, E5390, PR685, AIR2E and the reference sample by 31.78%, the peel forces for LT3366, SAER and PA1541 were significantly elevated (+ 172.97%). An exemplary analysis can be seen in Figure 5, where the Force-Time-Diagram for PA1541 on an untreated AlMg3 tool can be seen (50N consolidation force). When comparing the three different powder binders it is assumed, that the particle size has an influence on the adhesive behavior and that large particles have a beneficial impact. However, this needs to be studied in detail and the statement needs to be consolidated by additional microscopic analysis of the creep behavior.

The influence of AlMg3, V2A and composite tooling surfaces was studied for both untreated and pretreated (F770NC) conditions in Figure 7 (b) and Table 3. Only SAER and the prepreg reference sample have been considered for analysis. It is observed that in untreated conditions the tooling surfaces has no influence on SAER behavior, while F770NC application on CFRP tooling surfaces reduces peel forces by 70.53%. When looking at the reference prepreg samples, it can be stated, that only V2A substrate material significantly reduces the first ply adhesion (-63.49%). In general, it can be stated, that for both test series, AlMg3 tooling surfaces shows beneficial influence on peel forces (+51.97%).

Ultimately, the water-based release agent ZYVAX® 1070 W was compared against LOCTITE® FREKOTE 770-NC™ in Table 4. It can be seen, that Z1070W (-51.79%) shows less influence than F770NC pretreatment (-90.14%), when compared to an untreated AlMg3 surface.

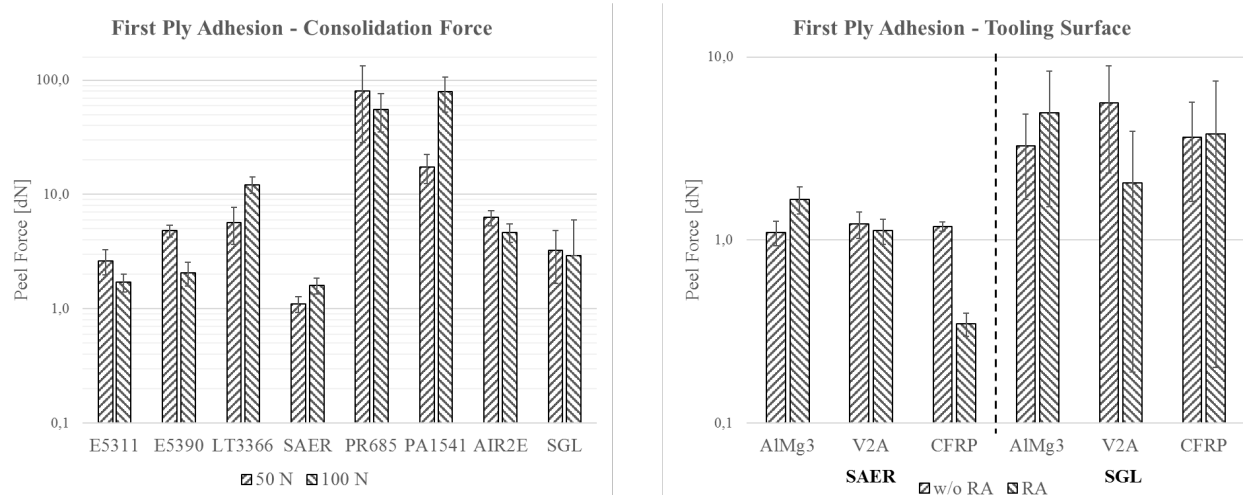


Figure 7: Influence on First Ply Adhesion (a) Consolidation Force (b) Tooling Surface

Table 2: Summary of First Ply Adhesion Measurements

Binder	Unit	Release Agent		Tooling Temperature		Consolidation Force	
		w/o RA	RA	RT	ET	50 N	100 N
E5311	[dN]	2,63 (±0,64)	0,42 (±0,19)	0,42 (±0,19)	0,41 (±0,02)	2,63 (±0,64)	1,71 (±0,31)
E5390	[dN]	4,83 (±0,58)	0,26 (±0,04)	0,26 (±0,04)		4,83 (±0,58)	2,06 (±0,49)
LT3366	[dN]	5,68 (±2,02)	1,69 (±0,35)	1,69 (±0,35)	1,00 (±0,41)	5,68 (±2,02)	12,20 (±2,06)
SAER	[dN]	1,10 (±0,17)	1,67 (±0,28)	1,67 (±0,28)	0,45 (±0,02)	1,10 (±0,17)	1,61 (±0,26)
PR685	[dN]	80,80 (±52,50)	21,13 (±5,31)	21,13 (±5,31)	2,76 (±1,96)	80,80 (±52,50)	55,67 (±20,40)
PA1541	[dN]	17,43 (±4,91)	1,72 (±0,43)	1,72 (±0,43)	0,97 (±0,51)	17,43 (±4,91)	79,77 (±26,89)
AIR2E	[dN]	6,27 (±0,93)	1,31 (±0,35)	1,31 (±0,35)	0,71 (±0,12)	6,27 (±0,93)	4,67 (±0,87)
SGL	[dN]	3,26 (±1,59)	2,65 (±1,41)	3,26 (±1,59)	1,50 (±0,44)	3,26 (±1,59)	2,93 (±3,03)

Table 3: Summary of First Ply Adhesion - Tooling Material

Binder	Unit	SAER		SGL	
		RA	w/o RA	RA	w/o RA
AlMg3	[dN]	1,09 (±0,17)	1,67 (±0,28)	3,26 (±1,59)	4,95 (±3,43)
V2A	[dN]	1,22 (±0,19)	1,12 (±0,0,18)	5,63 (±3,30)	2,06 (±1,87)
CFRP	[dN]	1,18 (±0,07)	0,35 (±0,0,5)	3,63 (±2,01)	3,79 (±3,59)

Table 4: Influence of Release Agents on First Ply Adhesion

Binder	Unit	Release Agent		
		w/o RA	Z1070W	F770NC
PA1541	[dN]	17,43 (±4,91)	8,40 (±4,32)	1,72 (±0,43)

Summary.

The first ply adhesion of seven different reactive and non-reactive tackifying agents have been analyzed and benchmarked against a pre-impregnated fabric according to DIN 28510-1. Various factors such as the influence of solvent- and water-based release agents, tooling materials, tooling temperatures and consolidation forces were taken into consideration and their suitability for DFP processes was analyzed. In general, it can be stated that surface treatment with a release agent has a negative impact on first ply adhesion (-77.78%) and that tooling surfaces at elevated temperatures will decrease the respective binder tack (-48.68%) accordingly. The influence of consolidation forces shows inconsistent results and needs to be further analyzed. When comparing different tooling surfaces, AlMg3 shows a beneficial impact on peel forces (+51.97%). Ultimately, it was shown, that water-based release agents have less impact on first ply adhesion, when compared to solvent-based release agents.

Concludingly, this study shows first insights on tackifying agents on overall composite performance, taking manufacturing technologies into account. Further studies on mechanical and thermal behavior are being conducted in order to get a comprehensive understanding on the influence of different reactive- and non-reactive binder systems.

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