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Experimental setup and method for the characterization of ply-ply adhesion for fiber-reinforced thermoplastics in melt

PIERIK Rens[1,2,a*], ROUWMAAT Thijs[1,2], GROUVE Wouter[1], WIJSKAMP Sebastiaan[2] and AKKERMAN Remko[1,2]

[1]Faculty of Engineering Technology, Chair of Production Technology, University of Twente, Drienerlolaan 5, Enschede, 7522 NB, the Netherlands

[2]ThermoPlastic composites Research Center (TPRC), Palatijn 15, Enschede, 7521 PN, the Netherlands

[a]e.r.pierik@utwente.nl

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Abstract. Process simulation software for hot press forming is a vital tool for the development of complex continuous fiber-reinforced thermoplastic parts for structural applications. The simulation tools need to be accurate to truly facilitate the design stage, which in turn requires accurate material characterizations and constitutive models. The material forming behavior is composed of different deformation mechanisms, one of which is the separation of adjacent plies or delamination. Currently, the resistance against delamination or ply-ply adhesion is modeled as a constant tensile stress that needs to be overcome, the value of which is based on an educated guess. To date, no standard exists to characterize this material property for thermoplastic matrix composites (TPC) in melt. Hence, we discuss and evaluate several methods to measure ply-ply adhesion of TPCs. The most promising approach, a so-called probe test, was further pursued and a setup was designed and manufactured for the use in a rheometer. Subsequently, we measured the required normal force to separate two C/LM-PAEK tapes in melt. Repeated tests on the same specimen resulted in an increasing adhesive peak force, which we relate to a change in the amount and distribution of the matrix material at the ply's surface. The peak force increased also with increasing compression time and pressure. We found a reasonable correlation of the average measured peak force with the values currently assumed in simulation software.

Introduction

Hot press forming is an attractive technology to manufacture small to medium-sized parts from continuous fiber reinforced thermoplastic blanks through remelting and reshaping. The process is rapid, cost-effective, and automatable. Complex parts can be produced with tailored lay-ups based on unidirectional (UD) fiber reinforced tapes using automated fiber placement (AFP) or automated tape laying (ATL) processes [1]. Moreover, thermoplastic matrix composite (TPC) parts can be welded to create large structural components [2]. However, the development of a proper production line becomes increasingly difficult with more complex parts and materials, as defects like forming-induced wrinkles could occur.

The development stage of a new part is facilitated by process simulation software that provide design engineers the tools for virtual design iterations and, with that, reduce trial-and-error costs [3,4]. The predictions of these simulations need to be accurate, especially on defect generation, to truly enable first-time-right defect-free manufacturing. In turn, accurate predictions require accurate material characterization and constitutive modeling to properly describe the material behavior during forming. Hence, different deformation mechanisms that occur during forming are

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commonly considered, which can then be characterized and modeled separately to capture the material's forming behavior.

One of the considered deformation mechanisms is delamination [5], which describes the separation of adjacent plies during the hot forming process. The resistance against delamination, or the ply-ply adhesion, is currently often described as a constant tensile stress based on an educated guess [3] or as a certain adhesion stiffness [6]. Moreover, the adhesive tension (normal direction) as used in numerical simulations indirectly affects the friction between adjacent plies (tangential direction) through the use of a penalty method that models the normal pressure in the ply-ply contact [5]. In our recent work [7,8], we investigated the ply-ply friction response to improve the constitutive friction modeling, for which the coupling with ply-ply adhesion of TPC in melt needs to be investigated in more detail as well. A standard to characterize the ply-ply adhesion for TPC in melt is to the best of our knowledge not defined.

The study of Mulye et al. [9] seems to be the only one that dealt with characterizing the ply-ply adhesion of TPCs in melt. These authors used a DMA with only the molten matrix material to measure the polymer melt strength as function of the debonding rate, which was subsequently used in a forming simulation for a fiber-reinforced PA-66 part to improve the prediction of local discontinuous plies (patches). Although the prediction of the patch's position and orientation improved, characterization of only the matrix material itself could result in an overestimation of the ply-ply adhesion, especially for plies with high fiber volume fractions. Contrary, the fibers were included in studies on the characterization of ply-ply adhesion of thermoset composites [e.g. 10-13], using different methods.

Given the above, this research aims to develop a setup and method to measure the required normal force to separate TPC tapes in melt for the characterization of ply-ply adhesion.

Background

The mechanisms and factors influencing ply-ply adhesion of TPCs will be presented in this section together with a brief description of some experimental methods to measure adhesion.

Ply-ply adhesion.

The creation of a bond between two molten TPC plies that are brought into contact develops through two mechanisms [14,15]. First, intimate contact or sufficient wetting needs to be established, meaning that the polymer chains of both surfaces are in close physical contact. Secondly, at locations with intimate contact, the polymer chains interdiffuse over the interface to form an entangled network, a process referred to as healing.

The formation of intimate contact is hindered due to the asperities on the ply's surface. Hence, the degree of intimate contact can be enhanced by applying a normal pressure. A lower viscosity of the polymer also promotes intimate contact formation. Thus, the time required for intimate contact depends on the applied pressure and polymer viscosity. The presence of fibers at the surface leads to local dry fiber-fiber contacts, which do not contribute to the formation of a strong bond. Therefore, Çelik et al. [16] proposed an effective intimate contact, considering only the intimate contact of matrix-rich areas in a study on contact development of UD C/PEKK tapes with AFP. Squeeze flow and percolation of matrix material increase the effective intimate contact.

The healing process starts once the polymer surfaces are locally in intimate contact. The polymer chains diffuse and form entanglements across the interface, leading to a fully entangled network comparable to the one in the bulk. The time required to establish this state depends on the polymer dynamics, for which generally de Gennes' theory of reptation is used [17]. A typical time in this theory is the reptation time, which denotes the required time for a certain polymer chain to escape from its initial configuration and, with that, form new entanglements with neighboring chains. According to Wool et al. [14], the strength of an interface grows through interdiffusion with time to the power ¼ and a fully entangled structure is achieved at a timescale comparable to

the reptation time, which is probably in the order of less than a second for the polymer melt considered here [18].

The timescales involved in ply-ply bonding of TPCs are, however, not so straightforward, as experimental studies found hundreds of seconds for healing [15,19] or intimate contact formation [20]. Polymer degradation, reducing the chain mobility, could have affected the healing process in the study of Avenet et al. [15], while the relatively long timespan for intimate contact formation found by Levy et al. [20] is probably due to the low consolidation pressure applied (1.34 kPa).

Adhesion test setups.

Adhesion was frequently characterized for thermoset composites for a variety of purposes as well as for the performance of (pressure-sensitive) adhesives. An overview of the different methods found in literature is visualized in Fig. 1 [21,22]. Crossley et al. [22] designed a setup to peel prepreg tape off a surface (Fig. 1a). A comparable test method is the T-peel test (ASTM D1876) [23], the fixed arm peel test [23], and the floating roller peel test (ASTM D3167), as visualized in Fig. 1b-d, respectively. For all four tests, the adhesion is quantified by gradually peeling surfaces from each other. Other techniques for adhesion characterization are the probe [10,12] and loop test (ASTM D6195), schematically illustrated in Fig. 1e and 1f, respectively. An interface is created by bringing the surfaces into contact and the formed adhesion can subsequently be measured by pulling. The probe and loop test are based on a more direct separation of the surfaces in normal direction, though the loop test actually features a combination of peeling and pulling.

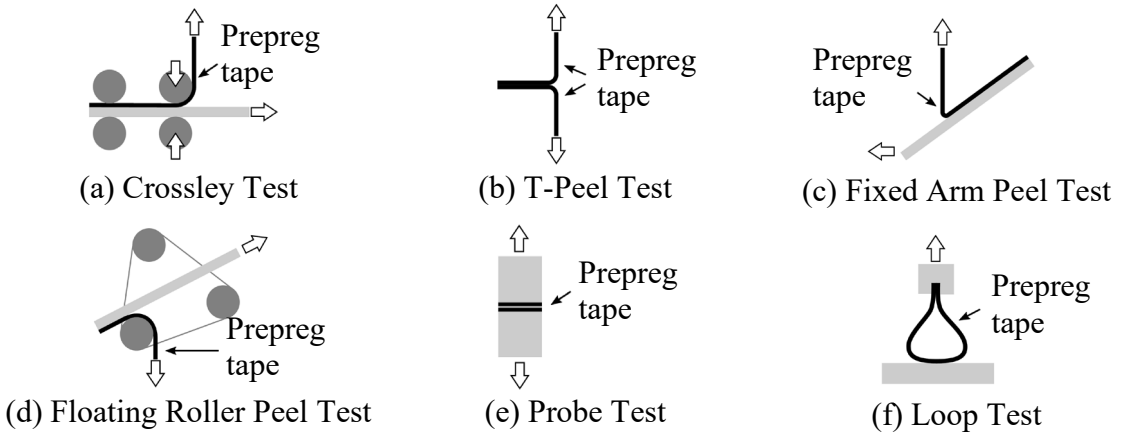

Fig. 1. Schematic illustration of several testing methods to measure adhesion [21,22].

Design of the Test Setup

A brief concept evaluation will be presented first followed by the final design of the test setup.

Concept evaluation.

From the four peel methods (Fig. 1a-d), we expect that the T-peel test (Fig. 1b) would suit the best for the use with TPCs, as it avoids the use of moving or rolling parts and the specimen can be pre-consolidated to form the ply-ply bond, closely resembling the situation in actual hot press forming. Contrary, the formed contact with the loop test (Fig. 1f) is not predefined and the stiffness and geometry of the loop will affect the measurement. The probe test will be more robust regarding contact formation and probably results in a more straightforward separation by pulling compared to the loop test.

Preliminary experiments were conducted with both the T-peel and probe method to determine the best suitable approach. During testing with the T-peel method, unwanted movement of the specimen due to slight misalignments and/or the convective heat flow in the oven resulted in variations in the pull force. Further, this measured force is a combination of adhesive failure and

bending of the specimen. Contrary, the pull force measured with the probe test can directly be used to calculate an apparent tensile stress for adhesive failure, as considered in simulation software for hot press forming. The probe tests was also slightly easier regarding specimen mounting and test cycle time. However, we observed slippage of the specimen in the clamps during the probe tests and specimens tended to bend in the force direction. These effects need to be minimized to reduce variations and to obtain a pure ply-ply adhesion measurement. All in all, the probe test was found the most promising.

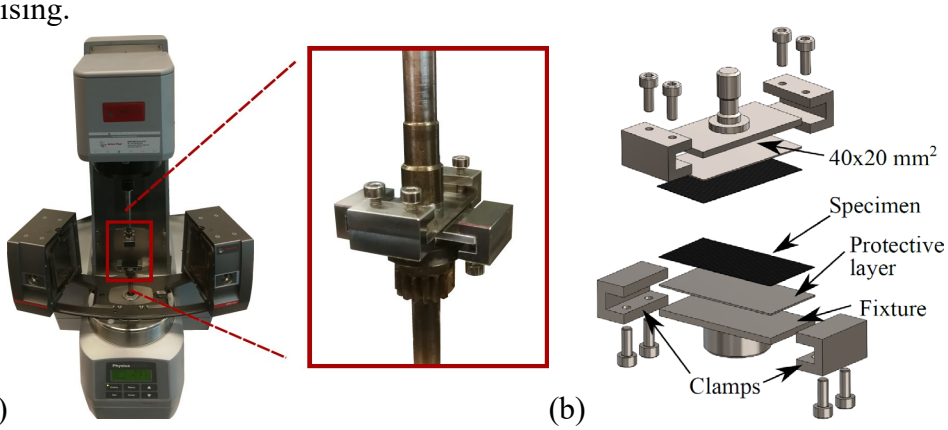

Fig. 2. Test setup mounted in an Anton Paar MCR501 rheometer (a) and (b) exploded view of the test setup with top and bottom fixture featuring direct connections for the rheometer.

Setup design.

The probe test fixture as used for the preliminary tests was further developed. The final setup mounted in an Anton Paar MCR 501 rheometer is shown in Fig. 2a. An exploded view of the designed setup, consisting of a top and bottom fixture out of stainless steel with direct connections for the rheometer is shown in Fig. 2b. The clamps at either side of the fixtures can be used to fix the TPC tapes. The usable surface area of each fixture is 40x20 mm^2, resulting in an overlap area of 20x20 mm^2 in case of a 0°/90° interface. Protective layers out of steel, measuring 40x20x1 mm^3, were used to shield the fixture from the molten polymer and served as a backing material for the TPC tape (see Fig. 2b), as will be discussed in the next section.

Experimental Work

Materials.

The composite material used in this research consisted of a unidirectional (UD) carbon-fiber reinforcement with a pre-impregnated LM-PAEK thermoplastic matrix. The tape, known as TC1225 C/LM-PAEK, was manufactured by Toray Advanced Composites. The melting temperature equals 305°C, according to the manufacturer.

Specimen preparation.

The tape material was supplied in 12 inch rolls, from which 39x18 mm^2 strips were cut with the fibers in the longitudinal direction. The strip width was chosen slightly smaller than the width of the fixture to ensure that the whole specimen was supported. The matrix material at the ends of the strips was removed using a blow torch to improve the clamping of the specimen in the setup. Next, a co-consolidation step was used to bond a strip to the protective metal plate. A bond between the metal plate and the tape material stronger than the ply-ply adhesion avoids parasitic bending of the specimen during testing, i.e. the metal served as a rigid backing material. Strips were placed on the protective layers in an oven at a temperature of 330°C under a slight pressure (around 50 kPa) for 10 minutes. The specimens were dried overnight before testing.

Test procedure.

Two co-consolidated strips were clamped in the top and bottom fixture of the test setup (see Fig. 2b) to form a test specimen. The fixtures were then mounted in an Anton Paar MCR501 rheometer and the oven was set to the test temperature T_t. A nitrogen flow was applied to avoid degradation during testing. A schematic illustration of the experimental procedure is shown in Fig. 3. The tapes at the top and bottom fixture were brought into contact with a controlled compression force F_c, resulting a normal pressure p, for a compression time t_c. After t_c, the force was put to zero for a short period of time t_x to ensure a force reading equal to zero at the start of the measurement. Subsequently, a constant upward displacement rate $\dot{\lambda}$ was applied to the top fixture to separate the plies, yielding a peak force F_{max}, as illustrated in Fig. 3. Besides the force, the time t and displacement λ were logged as well with a sampling frequency of 200 Hz. The measured data was smoothed with a Gaussian filter with a 50 points window to suppress the high-frequency scattering.

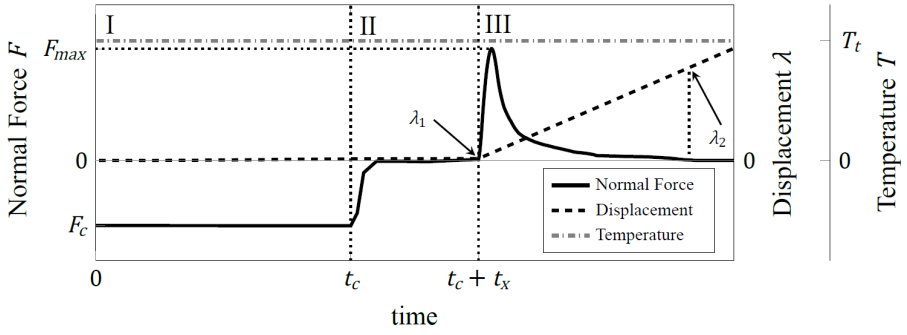

Fig. 3. Experimental procedure used to measure ply-ply adhesion.

Experiments.

All tests were performed at a temperature of 340°C and a displacement rate of 0.05 mm/s. In a first series of experiments, the effect of repeated testing of the same specimen on the peak force was investigated. Repeated tests on the same specimens, under different conditions, greatly reduces the experimental time required for a full characterization of the ply-ply adhesion. A normal pressure of 15 kPa was applied for 120 s followed by separation of the plies, which was repeated for ten times and performed for three specimens.

In a second series of tests, we investigated the effect of the compression phase on the subsequent ply-ply adhesion force by varying the compression time and pressure as listed in Table 1. The measurements were conducted in triplicate and a fresh specimen was used for each test. The longer compression times marked with an asterisk were only measured with a pressure of 15 kPa.

*Table 1. Test matrix for the second series of tests to investigate the effect of the compression phase. Compression times marked with * were tested only with a pressure of 15 kPa.*

Parameter	Input	Unit
T	340	°C
$\dot{\lambda}$	0.05	mm/s
p	3, 15, 31	kPa
t_c	10, 30, 60, 120*, 240*, 480*	s

Microscopy.

The ply's surface in the contact area was analyzed using a Keyence VHX-7000 digital microscope before and after testing to investigate whether the test invoked changes in the surface morphology of the tape. Micrographs were made from tapes in pristine state and after being measured for 3 and 12 times.

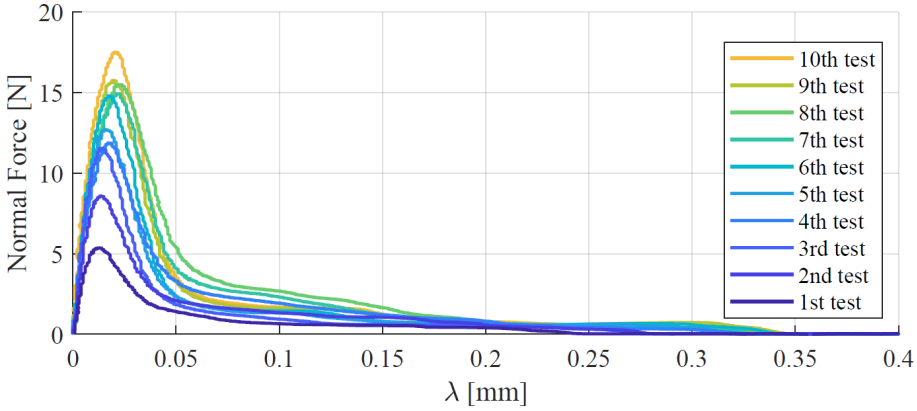

Fig. 4. Normal force versus displacement curves for a single specimen measured ten times with a compression phase (time of 120 s and pressure of 15 kPa) between each run.

Results

The normal force-displacement curves of a specimen tested repeatedly for 10 times are shown in Fig. 4. A clear peak adhesion can be observed followed by a gradual decrease in force towards zero at full separation around 0.3 mm. The peak force F_{max} gradually increases with increasing number of repetitions. The average trend of F_{max} over three specimens as function of the number of tests is shown in Fig. 5a. The error bars denote the standard deviation, indicating a large spread between the specimens. The increase in F_{max} with succeeding tests is further visualized in Fig. 5b, in which F_{max} is normalized with the first measured peak force for each of the three specimens.

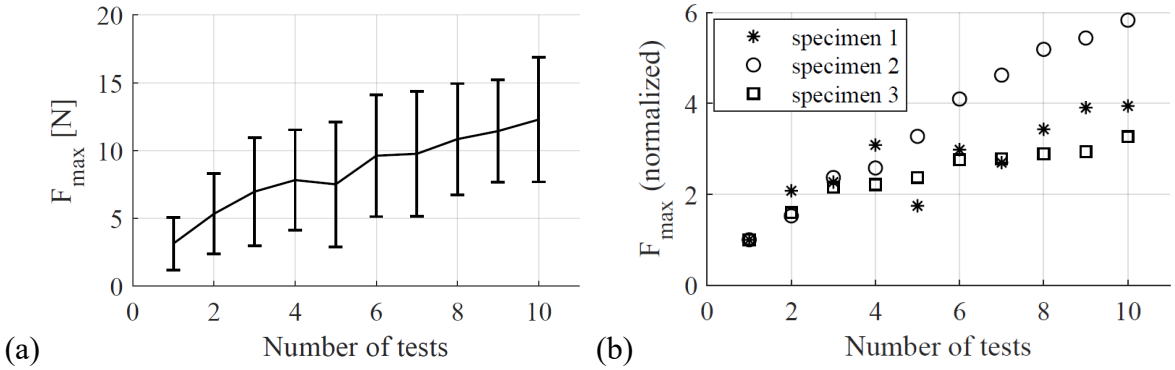

Fig. 5. Average peak force with number of repetitions (a) with the error bars denoting the standard deviation over three specimens and (b) normalized peak force with repetitions for each of the thee specimens measured.

The effect of the compression phase on the measured peak force is shown in Fig. 6a and 6b through varying the compression time and applied pressure, respectively. A longer compression time or higher pressure resulted in a higher F_{max}. Within the chosen experimental window, the peak force continued to grow without showing a sign of an asymptotic value. Even for the tests conducted with an extended compression time at a pressure of 15 kPa (see Fig. 6a), a limiting value for the peak force was not reached.

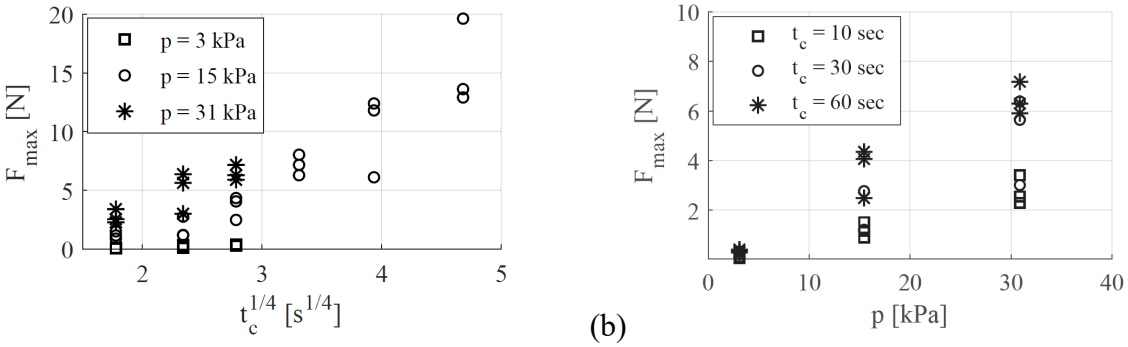

Fig. 6. Effect of the compression phase on the measured normal peak force through (a) the compression time and (b) the applied pressure.

Micrographs of the surfaces of the plies before and after testing are visualized in Fig. 7. The pristine state, shown in Fig 7a, clearly shows the fiber direction with matrix material distributed over the whole surface, shown by the lighter shade areas in the micrograph. The matrix material tended to form clutters in the contact area on the ply's surface after three repeated adhesion tests, as visualized in Fig. 7b. We observed a partition on the ply's surface, as highlighted in the micrograph by the red line, with contact (left) and non-contact (right). With more repetitions, the clutters formed into larger patches of matrix material, as visualized in the micrograph of a ply's surface tested for 12 times (see Fig. 7c). Note that the micrographs only show a small part at the boundary of the ply-ply contact, though the observations outlined above a more general and apply to the whole contact area.

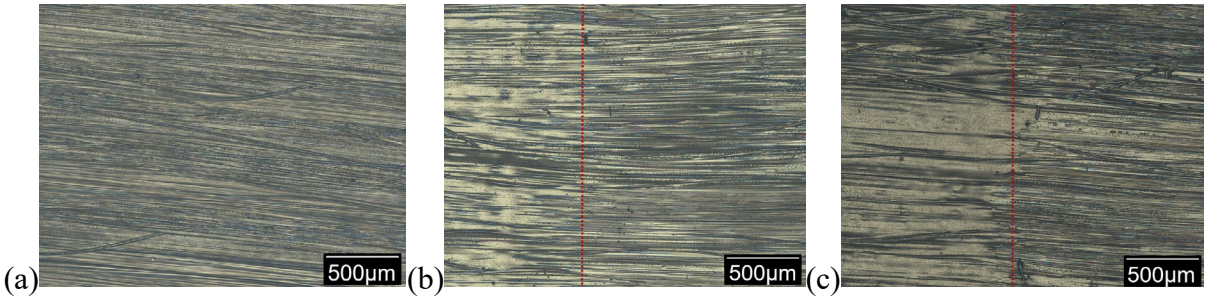

Fig. 7. Micrographs of the ply's surface (a) in pristine state and after tested for (b) 3 times and (c) 12 times. The red line approximately corresponds to the border between contact (left) and non-contact (right). The matrix material is visible by the lighter shade color.

Discussion

Repeated tests.

The peak force increased with the number of repetitions on the same specimen (see Fig. 5), meaning that reconsolidation of a specimen in the setup affects the subsequent ply-ply adhesion. The change in adhesion after repeated reconsolidation suggests to measure a specimen only once, consequently increasing the experimental time required for a full characterization of the ply-ply adhesion. More importantly, this reconsolidation effect disputes the representativity of the initial consolidation to simulate the actual ply-ply bond during hot press forming. Based on the experimental observations and the micrographs of the ply's surface as shown in Fig. 7, we believe that the force increase relates to a change in the amount and distribution of the matrix material in the contact area after repeated testing. The formation of clutters and larger patches of matrix material may be due to polymer strings between the top and bottom ply. These polymer strings tend to pull matrix material towards the ply's surface during a ply-ply adhesion test. Once a polymer string fails, the polymer settles in clutters on the ply's surface. With more repetitions, more matrix material gathers at the surface, forming larger agglomerates and increasing the

subsequent peak adhesive force. A change in the matrix material itself through degradation was found less probable to explain the increase in ply-ply adhesion based on control tests without nitrogen and DSC measurements.

Besides the increase in adhesion with repeated testing, quite a large spread was obtained on the measured peak force F_{max} as indicated by the error bars in Fig. 5a. This variation could be attributed to local variations in the fiber volume fraction at the ply's surface, leading to different effective intimate contacts between the specimens. Preliminary tests with an additional layer of matrix material in the ply-ply interface substantiated this idea of a change in effective intimate contact, as we measured a lower spread in F_{max} (not shown here).

Effect of the compression phase.

In the second series of tests, the effect of the compression phase on the measured peak force was investigated using fresh specimens for each test (see Fig. 6). The peak force increased with increasing compression time and pressure, as expected from literature. A higher pressure enhances the formation of effective intimate contact, resulting in a higher adhesion [15,16]. The timespan at which we observed an increase in ply-ply adhesion is also comparable to findings in earlier research on TPCs [15,19,20]. It remains unclear which mechanism, either intimate contact formation or healing, dominates this behavior. A ¼ power relation between force and time seems to appear at longer compression times, suggesting a healing process, though more experiments are required to validate this trend.

An apparent tensile stress σ_{max} can be computed through dividing the measured peak force by the total surface area of 18x18 mm^2, which can be used in process simulation software to describe the ply-ply adhesion. The maximum apparent tension values for different compression times and pressures are listed in Table 2, which are of the same order of magnitude as considered elsewhere. For example, Haanappel [5] estimated an adhesive tensile stress of 100 kPa for forming simulations with C/PEEK using the software package AniForm [4]. Recently, the tensile stress was lowered in an update on AniForm based on advancing insight. Lastly, Mulye et al. [9] found an average tensile stress of 6.2 kPa in their research on a thermoplastic composite with a PA-66 matrix.

The compression phase in the ply-ply adhesion tests was necessary to create a bond between the two ply's surfaces that were brought into contact. Ideally, the created ply-ply bond mimics the one present in pre-consolidated blanks or in layups produced with AFP or ATL to properly simulate the actual hot press forming process. However, the exact time and pressure that should be used for the compression phase remains unclear. Future research could be to investigate which values for the compression time and pressure should be used to mimic a certain process or the use of pre-consolidated specimens could be considered.

Table 2. Maximum apparent tensile stress σ_{max} in ply-ply adhesion for different compression times t_c and pressures p computed from the measured peak force and the surface area.

σ_{max} [kPa]		t_c [s]					
		10	30	60	120	240	480
p [kPa]	3	0.3	0.7	1.1	-	-	-
	15	3.7	5.3	11.2	22.1	31.2	47.5
	31	8.5	15.5	19.9	-	-	-

Summary

A setup was designed and manufactured to measure the required normal force to separate two plies, or the ply-ply adhesion, of thermoplastic matrix composites (TPC) in melt. The design is based on a so-called probe test, as used in adhesion tests for thermoset composites, and can be mounted in a rheometer. TPC tapes were brought into contact for a certain compression time under

a controlled pressure, after which the formed bond was separated with a constant upward displacement rate while measuring the normal force. Repeated adhesion tests on a single specimen with a compression phase between each run resulted in an increase of the normal peak force, implying that a fresh specimen needs to be used for proper ply-ply adhesion characterization. The increase in force was attributed to the formation of clutters and patches of matrix material in the contact area on the ply's surface, increasing the peak force after each adhesion test.

Increasing the compression time and applied pressure resulted in an increase of the peak force. A higher pressure is expected to increase the formation of intimate contact, consequently increasing the ply-ply adhesion. The compression time could affect the peak force through both the formation of intimate contact and the healing process. The latter mechanism might prevail as the force versus time curve seems to follow the typical ¼ power law relation, though the data is not conclusive enough on this aspect. Future work is to determine suitable parameters for the compression phase to mimic the ply-ply bond as formed in pre-consolidated blanks or after the AFP or ATL process.

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Data availability
The supplementary data for this conference article is available in the 4TU.ResearchData repository under DOI: 10.4121/21805422.

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