Tack measurement of bindered rovings for the dry fiber winding process

Stefan Neunkirchen | Ralf Schledjewski

Abstract
For the winding of dry (e.g., not impregnated) fibers on convex structures, an adhesive force between surface and roving has to be established. Providing friction is necessary to prevent the roving from slipping. Usually, rovings impregnated or coated with (epoxy-) binder material are used. The investigation of the tack performance of these rovings differs from established studies on pressure-sensitive adhesives and prepregs. It was found that the common probe test is not suitable for bindered rovings. Instead, shear tests were performed on a heated, flat plate with a robotic winding setup. The horizontal force to pull the roving from the plate was measured. Variation of temperature was in focus. Compaction force, pulling speed, and twisting of the roving were also investigated. It could be seen that compaction and thorough heating are the key factors. Due to the small surface, twisting had no significant effect on the tack behavior.

KEYWORDS
binder, dry fiber winding, probe test, shear test, tack

1 | INTRODUCTION
Filament winding is a well-established process for the high-volume manufacturing of mostly axisymmetric composite parts. Developments in automation, robotics, and simulation allow improved control of the process. In consequence, non-geodesic paths can increase the design freedom for new mandrels or more complex winding patterns on existing ones.[1,2] Within the frame of this paper, the dry fiber winding process will be regarded.[2] Here, dry or binder-impregnated rovings are used. For the filament winding of convex structures, the friction between roving and the wound-on surface plays a crucial role in the stability of the chosen paths. The friction coefficient determines the degree to which the desired path may deviate from the frictionless geodesic path.[2–4] Apart from mechanical fixation, for example, pins, the application of binder (also denoted as tackifier) materials is the common solution. Binders are usually thermoplastic or thermosetting (often epoxy-based) polymers that provide tackiness in a certain temperature range.[5] Since the activation is reversible, they are less sensitive to environmental influences compared to prepreg material. The concentration of binder on a bindered roving is relatively low (4–10 wt%) to enable a later liquid composite molding (LCM)-process.[6,7] Consequently, the ability to provide intimate contact between adhesive and surface is reduced. This also means that the existing test methods have to be adapted and the focus has to be laid differently.
1.1 Tack comparison: Binder, prepreg, and pressure-sensitive adhesives

Tack is an important parameter in the manufacturing of impregnated reinforcing materials. ISO 472 defines it as the "property of a material that enables it to form a bond immediately on contact with another surface, which can be an adherend or another layer of adhesive."[8] That is, it is a (experimentally determined) value for the stickiness/tackiness of a material. Yet, there is no standardized method for the determination of tack: it is highly dependent on the process, the material combination, and the expected loads. Simply touching the material with the thumb has been a hands-on testing method for a long time.[9] Starting with the investigation of pressure-sensitive adhesives (PSA), a scientific approach to determine the tack of materials has been established. In the manufacturing of composites, prepreg materials were first to be examined.[10–12] Starting from manual stacking, laying and placement processes in aerospace, like automated fiber placement (AFP) and automated tape laying (ATL), became more dominant. Since then, many studies on tack properties, measurement, and influencing parameters have been performed. The main findings relevant to this work will be presented shortly in the following. For a more detailed perspective on prepreg tack, the review by Budelmann et al. is recommended.[13]

Tack is a primarily viscoelastic property that is influenced by material, processing, and environmental conditions.[11] It is usually measured between two adherends. The surface may be of the same material but is usually metallic like the tool surface in the respective process. In particular, prepreg–prepreg bonding is up to 5.5 times stronger than prepreg-metal.[14] Bonding of the first layer is the most critical part in processes like AFP or ATL, especially when a release agent is used.[13,15]

Although many material combinations have been investigated in the past, only a sparse general understanding could be achieved.

PSA tests are easier to evaluate because the adhesive can be seen as a bulk. Only adhesive and cohesive forces have to be differentiated. Duncan[16] assumes that surface adhesion is a requirement but the cohesive properties determine the strength of the bond. Zosel states that the maximum tack can be found 60–70°C above the glass transition temperature, due to compliance and related to the entanglement network of the polymer.[17]

Compared to PSA, the analysis of prepregs is more complicated due to anisotropy, heterogeneity, fibers as part of the surface, and possible gradients of the resin content.[10,18] It is assumed that the behavior shifts from predominately viscous to elastic because of the fibers.[10] Also, the failure modes differ from those of PSA. Effects like cavitation and fibrillation are less dominant. Fibers prevent the occurrence of bulk polymer regions. Low-tack systems tend to experience interfacial failure with less resin leftovers. High-tack prepregs fail cohesively in the bulk with possible fibrillation. A transition between these modes can be observed with a change of temperature.[15]

Overviews of possible influencing parameters and conditions are given by Budelmann et al.[13] and Crossley et al.[15] Table 1 shows typical values for PSA and prepreg that have been studied in the past.

Temperature is an important factor in designing a process or testing method. While PSAs are usually used at room temperature, they were tested in a wide range to investigate all possible influences on the pure polymer. Prepregs are usually processed at temperatures slightly above room temperature, that is, 40–50°C. Here, premature degree of cure and required tack have to be balanced. Binder materials can be activated repeatedly at elevated temperatures, typically between 80 and 150°C. This means that a possible test setup for binder tack needs to provide and to withstand these temperatures. Below the melting temperature, no material response is expected.

Depending on the test type, equipment, and regarded parameters different compaction forces and dwell times are used. As Table 1 shows, a wide range has been tested in the past. In general, the compaction force is higher for prepregs. Here, the tests focus much more on industrial machine usage, whereas PSAs are often applied manually. This also explains the wide range of dwell time tested for PSA on the one hand and the tendency to very short times on the other hand. The test velocity is mainly determined by the given machinery, be it the limits of the test rig or the intended production speed. All of these parameters have yet to be determined for the testing of bindered rovings. Prepreg processing should be seen as the guiding principle. Although there is usually no compaction intended in the dry filament winding process (other than induced by the roving tension), it has to be applied in the testing environment.

For prepregs, much effort has been put into the investigation of the influence of relative humidity, degree of cure, and pot life/out-time. All these parameters are closely connected and can influence the AFP/ATP process and its outcome severely. Since binder material should not start curing during the process, this investigation is irrelevant. Nevertheless, it should be investigated in future studies to which degree small variations of the binder content or relative humidity influence the test results.

Other parameters that have been pointed out to influence tackiness are, for example, viscosity, (reinforcing) structure, defects, dirt, different testing surfaces, and
resin residues.[13,15] These affect any given material, so they will not be explored in this work. All test results apply only to one specific material. Consequently, the proposed testing method is only a recommendation for a specific material, process, environment, and the given equipment. To completely understand tack and its influences, fundamental research on surface wetting, polymer viscoelasticity, and polymer failure mechanisms is necessary.[13]

1.2 | Short review of tack testing

Several tack-testing methods have been used in research and industrial application. Many publications have presented an overview of the various testing strategies.[9,11,13,15] Tack is a property that strongly depends on the given process and material combination. Hence, the selection of the right testing strategy is important to receive suitable results.[9] The methods depicted in Figure 1 will be presented shortly and evaluated regarding their applicability for bindered rovings and the filament winding process.

1.2.1 | Probe

Originally developed for the testing of PSA, many methods have been adapted in the composite industry for the testing of prepregs. Probe tests have been standardized in the PSA industry[19] (withdrawn 2019 due to limited use in the industry). The working principle is the pressing of a stamp on the substrate and then measuring the force when separating it, for example, in a universal test rig. The main advantages are good reproducibility, the possibility to observe the failure mode, and flexibility regarding material choice and properties. Besides, many environmental conditions like heat or moisture can be simulated in adequate housings. Although planar structures like textiles or films are particularly suited for probe testing, tests on a roving level are also possible.[9–11,13,17,18,20,21]
Since probe tests are simple to perform yet reliable, they will be used first in the experimental section to get an overview of the behavior of bindered rovings.

1.2.2 | Peel

Peel tests are of special interest in the AFP/ATP-processes. Here, a balance has to be found between a maximal adhesion on the surface and a minimum on the compaction roller or backing paper. Tack is measured by peeling the substrate off a surface and recording the force. This can be a simple setup as depicted in Figure 1, a more advanced setup like the apparatus developed by Crossley et al., or the ASTM floating roller test. While the ASTM setup merely provides a robust and reproducible test environment with given angles and loads, Crossley’s setup adds a compaction load and simultaneous movement of both parts. Compared to probe tests, peel tests lack reliability: the mode of application is more difficult to standardize and the failure modes and stages are harder to observe.

Regarding bindered rovings in filament winding, peel testing is no option. Apart from a process like tape winding, there is commonly no compaction roller involved and hence no possible peel force acting on the roving. In addition, the adhesive film on a bindered roving is less strong and uniform than on a prepreg so the expected forces and failure modes may not be displayed correctly in the peel setup.

1.2.3 | Loop

Loop tack tests are predominantly used for PSAs. Loops of adhesive tapes are pressed on a rigid surface and then separated. Forming the loop evenly and applying the pressure on a defined area are just two major inaccuracies of this process. Therefore, loop tests are not considered for the bindered rovings (and have not been for prepregs yet).

1.2.4 | Rolling ball

The rolling ball test is a very simple test to estimate the tackiness of a material. A ball is rolled from a ramp onto an adhesive tape. Contrary to the force measurement of the other methods, the distance the ball covers is recorded.

This method can be used to get an impression of a material’s self-adhesiveness but is not suited for scientific investigations of tack.

1.2.5 | Online

Böckl et al. proposed a setup for monitoring the tack of prepregs in the AFP process. While moving, the slit tape will be pressed against a three-axis load cell and the transverse force is recorded. Although experiments showed some promising results, this setup is not fully developed and needs to be validated.

1.3 | Friction coefficient/slippage tendency

The measurement of the friction coefficient is an established method in filament winding. Here, the required friction that prevents the roving from slipping off a non-geodesic path is determined. Koussios et al. proposed a special mandrel shape that allows a linear relationship between friction coefficient and machine feed.

With bindered rovings, this friction force can be provided by the tackiness of the binder. However, the availability of a (uniformly) heated mandrel is a major restriction.

Therefore, simpler tack experiments should be used to determine the parameters for the maximum tackiness of a bindered roving. These can then be implemented in the filament winding environment to provide optimal processing conditions.

2 | EXPERIMENTAL

Many testing methods have been developed to describe tackiness. Only a few of them can also be applied to bindered rovings. The first choice for the experimental part will be the easy and reliable probe test. Based on the findings in these experiments, a new shear tack testing method will be proposed. The rovings will be placed on an even surface and then pulled off. A quite similar approach was already used to manufacture lap shear test samples using an AFP process.

2.1 | Material

Binder materials are available in a variety of forms: powder, spray, veil, or hot-melt liquids. The most widespread material classes are thermoplastics and epoxy resins without the B-stage curing portion. Many applications of wound parts require the use of epoxy resin to provide the requested mechanical properties. To the authors’ best knowledge, only one epoxy-bindered roving is commercially available.
in Europe: Tenax-E HTS 40 X030 by Toho Tenax/Teijin. This 12 k carbon fiber roving with a density of 800 tex is coated with approximately 7 wt% of epoxy resin. According to the data sheet, the binder should be activated at approximately 120°C but not exceed 160°C to prevent curing of the material.\(^{[33]}\)

A test-rig for the application of powder binder on rovings or tapes is currently being set up to enable flexible manufacturing of bindered rovings with varying materials and amounts.

### 2.2 | Probe tests

Probe type tack tests are still standard for the measurement of adhesive properties due to their easy setup and good repeatability. Despite a different load case than in the winding process, this fast and reliable testing method is expected to give a good overview of the adhesive behavior of bindered rovings. Therefore, pretrails should evaluate if probe tests are suitable for this material. The primary aim is the determination of the optimum temperature window for the activation of the binder. For this purpose, a compaction test setup on a universal testing machine was used. It is designed for the measurement of the compaction and relaxation of laminate stacks under temperature influence up to 250°C. A 30 kN load cell was used. To enlarge the contact area on the stamp (100 mm diameter) and raise the measured tack force, five rovings were placed in parallel. In accordance with,\(^{[34]}\) a compaction pressure of 10 kN was held for 5 s until the plates were separated with a speed of 1 mm/s. The maximum negative deflection was recorded and evaluated for different temperatures. At least five tests per temperature have been performed to provide statistical validity.

### 2.3 | Shear tests

In addition to probe tests, a simple and repeatable test routine for tack measurements of bindered rovings shall be defined. It needs to be designed according to the setup of the desired manufacturing process, that is, dry fiber winding. Also, the load case in the test should be comparable to the actual winding process. Vertical loads will lead to fiber bridging or peeling and must be avoided in a winding process. Hence, a load tangential to the mandrel’s surface can be assumed. Shear tests are a well-established method to characterize adhesive bonds although the load case is usually not completely accurate (e.g., nonuniformity of the adhesive layer).\(^{[35]}\)

When processing bindered rovings heat has to be provided to activate the binder. A uniform and reliable heating of the mandrel is an enormous challenge for the process/testing equipment. This means that these mandrels are more expensive and often very specialized.

In consequence, a flat heating plate was chosen due to the easy availability and the acting shear loads during a test. Since no curvature is given on a flat plate, the only load acting is tangential to the mandrel surface due to the roving tension. Figure 2 shows the shear load \(\tau\) in the proposed setup. Since the roving contact surface will remain nearly constant, the roving tension is measured to determine the acting shear load of the bonding area between binder and surface. To provide contact between roving and plate, pressure has to be applied in the form of a weighted roller (see Section 3.2.2).

Especially, the transversal parts of this shear load lead to slippage on a convex surface and must be supported by friction forces.\(^{[2]}\) In this study, only a 0°-load (pulling direction) will be regarded because the highest force can be expected. Also, this setup is simpler and more transferable. Considering roving slippage on curved surfaces, the winding angle needs to be changed and further tests need to be conducted on a special heated mandrel.

The experiments were conducted with robotic filament winding equipment. Figure 3 shows the setup before a test. The roving was placed on an aluminum heating plate over the length of 1 m. This relatively large contact area shall ensure a good bonding quality and be easily comparable to more complicated geometries. Surface temperature was measured by a thermocouple fixed on the plate’s surface with polyimide tape. After a straight and even placement, two compaction rolls—weighted by an aluminum profile—to be able to alternate the compaction load were moved over the roving.

The robot performed a linear motion of 300 mm away from the plate after starting the experiment. Fiber guidance in the winding head was modified so that all the moving elements are fixed during the experiment. A load cell on one of the guiding rolls measured the maximum fiber tension as an indicator of the tack force. For each parameter variation, 10 experiments were performed to provide statistical accuracy. After each experiment, the surface was cleaned with mold cleaner and additionally with isopropanol to ensure degreasing.
RESULTS AND DISCUSSION

3.1 Probe tests

Probe tack tests were performed to evaluate the testing method and to determine the temperature window to activate the binder. Figure 4 shows that up to ~100°C the measured tack forces behave as expected; however, below 85°C, no significant forces can be detected. The binder remains solid. With the melting of the binder, the tack force rises to its maximum at 100°C. After that, the values drop again and then remain on a lower level.

Figure 5 shows differential scanning calorimetry (DSC) results. The melting temperature is determined to be at approximately 100°C. In the melting area, the results from the tack experiments correspond very well to those of the DSC.

At 120°C, the binder is fully melted and a different tack response can be seen. An effect that explains the high variation of the shown data is the spreading of the roving in vertical direction as shown in Figure 6. This behavior could be observed at different temperature levels and to different degrees, for example, adhesion to one or both plates and different “opening” distances of the roving. For example, the test series at 120°C degrees showed results of 22 N with little spreading of the roving and 7 N for a complete unfolding.

Hence, it must be concluded that the probe test is not suitable for single layers of bindered rovings. Contrary to Ahn,[10] who could model and test a prepreg stack as a bulk material, the mass percentage of the binder is only a one-digit value. Consequently, the distribution of the binder powder in manufacturing will be concentrated at the surface.

Figure 7 compares the investigated adhesive materials in this study. PSA and prepregs can provide a sufficient wetting of the surface with just scattered dry spots. Binder particles are concentrated on the surface of the
roving where they are intended to create an adhesive film to enable bonding. Yet, it is not possible to predict how these particles will behave after melting. They can contribute to the desired film on the outside but may also migrate inside the filaments and impregnate the roving. Consequently, the share of binder that is located between the filaments of the roving cannot easily be determined.

Additional studies have to be conducted to investigate the impregnation behavior of binder depending on temperature, compaction/tension, fiber volume fraction, particle size, and roving type. Therefore, the behavior of the roving under vertical load cannot be estimated reliably, that is, if and how much of it will fan out.

Concerning the winding process, a vertical load should never act on the roving. Therefore, the risk of uncontrolled spreading is not given. Nevertheless, this relatively easy tack test is not suitable for bindered rovings and an alternative has to be chosen.

### 3.2 Shear tests

Shear tack tests were performed to establish a testing method for the dry fiber winding process. The influence of four parameters in this setup was investigated.

![Shear tack force over temperature](Color figure can be viewed at wileyonlinelibrary.com)

#### 3.2.1 Temperature

An optimized activation temperature of the binder to provide the highest tack is the primary aim for the dry fiber winding process. Apart from the determination of the process window, it should be evaluated if a certain temperature provides higher tack and should therefore be recommended.

Figure 8 displays the results of these experiments. In accordance with DSC data (cf. Figure 5), the onset for the melting area is about 80°C. No significant force is measured until then. The leap in tack force at ~93°C can also be explained with DSC data: Here, the curve toward the melting point peak increases rapidly, that is, melting accelerates fast.

Contradictory to the expectations based on the experiences with prepreg tack,[13] no bell-shaped behavior could be observed in tack force. The measured forces stay at a nearly constant level. This shows that if the binder is fully melted, the tack force almost instantly reaches a constant level. Considering process control, this is a pleasant observation: the process temperature can be varied in a rather large range provided that a minimum temperature for the complete melting of the binder is given.

A second and rather challenging result is that the adhesive behavior can vary strongly. Even in a testing environment (although it is close to the actual process), it is difficult to provide constant conditions. Especially, the
surface preparation seems to be critical. Not only is a surface in a production environment often scratched or damaged in another way, different (auxiliary) fluids and dirt can influence its properties as well (see also Figure 11(A)–(C)). On top of that, exact placement of the roving cannot be guaranteed as well. The roving usually is not uniformly placed on the bobbin. Possible defects can even be amplified by the fiber guidance. Consequently, it must be ensured that the accuracy of the winding equipment is sufficient and that the surface is well prepared and protected from further contamination during the process.

In summary, the required tack of bindered rovings can be provided in a wide temperature range but environmental conditions need to be kept constant to ensure good adhesion.

In the following experiments, a temperature of 112°C (+/− 1°C) was selected to provide the best results.

3.2.2 | Pressure

Contrary to tape laying/placement processes, there is no external compaction by a roller in the filament winding process. The force keeping the roving in place on the mandrel is provided only by the roving tension. It is strongly dependent on the geometry of the mandrel. Since the roving tension is the force to be measured in this setup, external pressure has to be applied. For a high-bonding strength, intimate contact and hence complete surface wetting is crucial. In consequence, a small two-wheeled wagon was constructed. The silicon rollers were taken from a tape-laying head. The top platform can then be supplemented with additional weights.

After the roving is laid on the plate, the wagon is rolled over it. No significant difference in force could be observed if it was compacted once or twice.

As can be seen in Figure 9, without compaction, almost no force could be observed; The roving lacks contact to the surface in large areas and hence no adhesion between surfaces can be established. Considering high loads, the tack force increases with additional compaction weight. This corresponds to the results for prepreg tack reported in literature (cf. Table 2 in ref. [13]). Since the contact time is relatively low, no maximum can be observed in the regarded range.

For further experiments, only the self-weight of the wagon is used. Next to the practical effect, the possibility of damaging the fiber with lateral movements is too high. Also, the effect of a higher compaction load on the results is negligible compared to the results of non-compacted rovings.

3.2.3 | Velocity

Another parameter that can influence the tack level of a bindered roving is the velocity at which the roving is pulled off the heating plate. Since this force is usually not occurring during the actual winding process, it is just an evaluation for the accuracy of the testing procedure. The standard velocity was chosen to be 0.05 m/s. It is then increased up to 2 m/s, the maximum speed of the robot. It must be noted, though, that this velocity most probably will not be reached due to the short testing movement. To measure at high velocities, it is also necessary to use a sufficient detection rate of the load cell because the regarded peaks are very small. A data sampling rate of 20 ms has proven to be convenient.

Figure 10 shows that the measured force increases slightly. In addition, the standard variation increases significantly.

This behavior could be explained by two observations. Firstly, the “whipping” noise of the roving losing adherence shows no interference with the tack force. Secondly, the highest tack forces could be measured in experiments in which the roving was fanned out as depicted in Figure 11.

It is assumed that the sound of the unsticking of the roving is dependent on the cohesive forces between roving and plate surface. For high tack values, it can be observed that not only the roving loses adherence to the
surface, its fibers are pulled apart as well. In these cases, the binder must also have created a good bonding between the filaments of the roving. However, it should be noted that this effect could not be repeated reliably with the same processing parameters. It is highly dependent on environmental influences like surface quality and absence of roving defects. The parts of the roving despoiled from the edge of the bobbin are especially susceptible to irregularities. In consequence, the rather low-testing velocity of 0.05 m/s is maintained.

3.2.4 | Twisting

Fiber damage is a big issue in the winding process, dry fibers in particular. The roving guidance system has to be optimized in that regard. Nevertheless, the roving quality can never be guaranteed to be uniform. As mentioned before, particularly the edges of the bobbin are often damaged by impact or just by slippage of the tensioned roving. Sometimes, the rovings are also already twisted on the bobbin. Hence, the influences of these roving torsions on adhesive behavior should be investigated.

Therefore, one resp. three torsions were induced manually before placing the roving on the plate. The standard parameters and procedures apply.

Figure 12 shows that the influence of torsion on the shear tack force is marginal. For a single torsion, the tack force is even higher than the reference but this can be seen as a measuring inaccuracy.

The results can be explained by the rather small width of the roving and the marginal reduction by the torsion. Also, the compaction force for this smaller area remains the same. The bonding strength there is probably higher than in the non-twisted regions.

4 | CONCLUSION

A fast and simple method was presented to characterize the tack properties of bindered rovings: shear tack testing. In comparison with the established probe method for prepregs or PSA, this test is adapted to the specifics of bindered rovings in a dry fiber filament winding process. Parameter studies have shown that the tack force of an activated binder remains on a high level once the binder is fully melted. It can be increased slightly by applying more compaction pressure or increasing the haul-off velocity. In the winding process, however, these two parameters can only be altered in a rather narrow range by winding parameters such as roving tension, mandrel geometry, and process speed. These parameters are often fixed by the given winding equipment and part design. If possible, their influence on the roving’s tackiness should be considered though. Induced roving defects by torsions could be neglected in the examined case due to the small roving diameter. Further investigations have to observe the behavior of the binder when melting on the roving’s surface. Also, other material combinations need to be tested for further validation of this testing routine and to investigate influences such as binder content and distribution.

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