The influence of consolidation on bending behaviour of molten unidirectional fiber reinforced thermoplastic tapes

J.H.W. ter Bogt
Supervisor: Ir. D. Brands

University of Twente, Faculty of Engineering Technology, Drienerlolaan 5, 7522 NB, Enschede, The Netherlands
Corresponding author: j.h.w.terbogt@student.utwente.nl

ABSTRACT: Bending experiments were performed on non-consolidated and consolidated specimens of single-layer unidirectional fiber reinforced thermoplastic composite in molten condition. The results exhibit distinctive bending behaviour. The consolidated specimens portray reduction in stiffness and a higher viscosity, compared to non-consolidated specimens. Micrographs of the specimens were created before, during and after bending. Consolidation influences the micro-structure of the material by making the fiber-matrix distribution more homogeneous, eliminating most of the surface roughness and removing nearly all voids. This influence remains visible after deconsolidation and bending. Currently, material models of bending behaviour used in simulations of stamp forming, are based on characterization experiments of non-consolidated specimens. However, consolidated laminates are the input of stamp forming. It is thus advised to review the material characterization of the bending deformation mechanism.

Key words: carbon fiber characterization, thermoplastic composite, bending, optical microscopy, consolidation

1 INTRODUCTION

Fiber reinforced composites are used to produce strong and lightweight products. This type of material consists of a fiber and a matrix, where the fiber is often made of carbon, glass or aramid. The matrix is either a thermosetting or thermoplastic polymer, that keeps all the fibers together. When applying these materials, high specific stiffness and strength can be achieved compared to metals. This makes this type of material suitable for the aviation and aerospace industry.

Thermoplastic matrices offer benefits over thermosetting matrices. Short cycle times can be achieved due to their ability to be used in stamp forming, making them cost effective for mass production. They also offers welding capabilities, which aids in reducing manufacturing costs [1]. Moreover, thermoplastics inherently have higher toughness and impact damage resistance compared to thermosets [2].

Application of thermoplastic composites in production is constrained by the complexity of handling this material. A production process that is already used in the aviation industry is stamp forming. In stamp forming, a pre-consolidated laminate is heated above the melting temperature (T_m) of the polymer, typically using an infrared oven [3], quickly transferred to a press, shaped using a set of molds and solidified at the mold temperature after which the part is released [4]. The stamp forming process is illustrated in Figure 1.

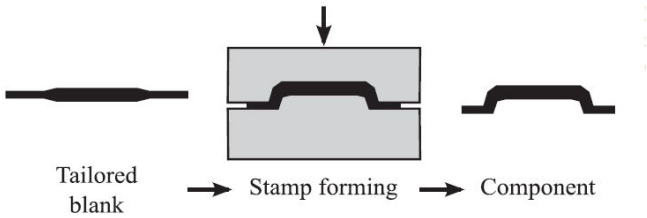


Fig. 1: Stamp forming process illustrated [5]

Stamp forming is time-efficient compared to conventional composite production methods. However, defects such as wrinkling occasionally occur during this process. These defects must be avoided as they negatively affect the mechanical performance and quality of the part. A simulation tool that can predict wrinkling and other (deformation) defects during the design phase of a part is therefore useful. However, the simulation tools for thermoplastic composite are relatively new, and need further research on material properties to be enhanced.

The simulations targeted in this research are based on finite element analyses. They take three governing deforming mechanisms into account: intra-ply shear, inter-ply and tool-ply slippage and bending [2]. To create sufficiently accurate modelling software, each of these mechanisms needs to be understood properly. Material behaviour is approached using material models based on characterization experiments. To make the simulation as accurate as possible, the characterization should be done with physical specimens with realistic influences taken into account.

During the heating phase of the stamp forming process, deconsolidation occurs as a result of high temperatures and lack of external pressure, visible in Figure 2. Deconsolidation is the process of lofting of a composite, which causes voids to form and grow within the composite, potentially causing plies to delaminate. A cause for deconsolidation that T. Slange et al. mention is the thermal expansion of dissolved gas and moisture [5]. Deconsolidation due to moisture expansion can be eliminated by drying the blanks.
The second cause for deconsolidation is the release of fiber bed stress [5]. Fiber bed stresses are introduced during the production of the thermoplastic composite. This stores elastic energy in the fibers, which is released upon heating causing the material to deconsolidate. Deconsolidation results in increased void content and poor interlaminar bonding, which could lead to subpar performance of the product [5].

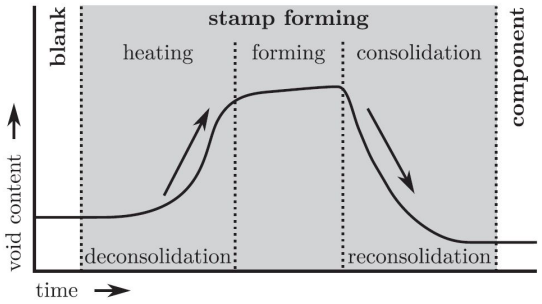


Fig. 2: Types of consolidation during stamp forming

According to Partou et al., deconsolidation is reduced considerably by consolidating the material, before shaping it [1]. To consolidate a thermoplastic composite, the material is heated above T_m under 10-20 bars of pressure for 10-20 minutes using a press [3]. Consolidation removes bubbles of air, improves the polymer distribution in between the fibers and may remove inconsistencies in the patterns of the fibers [1]. Consolidation also improves the interlaminar bonding.

When a part is to be made of thermoplastic composite, producers most often use layers of prepreg material. Prepregs are single-layers of composite made of fiber impregnated with a, in this case, thermoplastic polymer. Before production, a laminate is made by stacking multiple layers of prepreg material on top of each other, and consolidating the stack to form a laminate.

According to D. Brands, currently material properties for the bending deformation mechanism that are used in simulations for stamp forming, are based on characterization of bending of non-consolidated (dry) fiber reinforced thermoplastic tapes. However, the input of the stamp forming process is a pre-consolidated laminate. No prior research has been done on the influence of consolidation on the bending deformation mechanism. It is therefore necessary to investigate the influence of consolidation on bending behaviour of fiber reinforced thermoplastic prepregs. The investigation was done with a custom bending set-up on consolidated and dry single-layer prepregs of unidirectional (UD) fiber reinforced thermoplastic tapes in molten condition. A tape is considered UD when all of its fibers are oriented in the same direction. The goal of this research is to better understand this type of material and to give an advice on improvement of material characterization used in stamp forming simulations.

2 METHODOLOGY

Consolidated and non-consolidated specimens were prepared, tested using a rheometer incorporated in a custom bending set-up, and analysed.
Next to performing the bending experiments, the specimens were analysed using a Keyence VHX 7000 digital microscope to visualize the changes that happened on microscopic level during consolidation, deconsolidation and bending.

2.1 Materials

Two UD fiber reinforced thermoplastic materials will be investigated: Toray Cetex®TC1225 and Solvay APC (PEKK-FC). Both materials are single-layer

prepregs that consist of a carbon fiber, but use a different polymer as matrix. Toray uses a low-melt (LM) PAEK resin [6] whereas Solvay uses a PEKK-FC polymer [7].

Two materials by two different suppliers are used to be able to gain an understanding of variances in microscopic structure of this type of material, and to be able to better confirm the reliability of the results of the bending experiments. Toray Cetex®TC1225 and Solvay APC (PEKK-FC) are referred to as 'Toray' and 'Solvay' respectively in this paper.

2.2 Preparation bending experiments

Of both materials, dry and consolidated tapes need to be prepared. The preparation processes are described below.

2.2.a Consolidation

The consolidated tapes need to undergo a consolidation cycle before being bent. Toray and Solvay require different consolidation cycles, which are presented in Table 1.

Table 1: Consolidation cycle settings for Toray and Solvay

	Toray	*Solvay*
Pressure pre heat (bar)	2	2
Pressure main stage (bar)	15	10
Time pre heat (min)	10	10
Time main stage (min)	30	15
Temperature (°C)	365	375
Heating gradient (°C/min)	10	10
Cooling gradient (°C/min)	5	5

2.2.b Drying

Both Toray and Solvay are taken directly from a roll. This is referred to as 'as received' (AR). A minimal water content in the specimens is desired as humidity stimulates deconsolidation according to research of T. Slange et al [5]. The AR specimens are therefore dried in a vacuum oven at 70°C for at least 12 hours. After drying the AR specimens in a drying oven, the condition of the specimen is referred to as 'dry'. The time between retrieving the specimens from the oven and bending the specimens was kept as minimal as possible, and did not exceed 4 hours.

The specimens that are to be consolidated do not need to be dried before being consolidated, as consolidation removes humidity in the specimen. To make sure the consolidated tapes did not take up moisture in between the consolidation process and bending experiments, the specimens were stored in the drying oven.

2.2.c Dimensions specimens

The rheometer set-up requires the specimens to be of dimension 35x25mm^2, with the direction of the fibers along its longest side. This was done using a paper cutter and checked using a caliper. The thickness of every specimen was measured using a micrometer.

2.2.d Polyimide tape

To prevent contact between the specimen and the fixtures of the bending set-up, polyimide tape is attached to both ends of the specimen. This tape is heat-resistant and minimizes friction between the metal fixture and specimen [2].

2.3 Bending experiment

The preparation steps of the bending experiment are described in this chapter.

2.3.a Set-up

The experiments were performed using a custom-built bending set-up, which has previously been developed at the University of Twente [2]. The set-up is visualized in Figure 3. An Anton Paar rheometer MCR 501 combined with a thermal chamber CTD 450 performs the bend on the UD thermoplastic tapes. The specimens are placed in position with fibers in the direction of their length.

The rheometer measures the required bending moment applied on the tape (M_c) at a deflection angle, given a rotational velocity. The tape is held in position by custom made steel fixtures with minimal force to allow sliding in horizontal direction [2]. The specimens rest on two support pins.

The size of clearance c in Figure 4 should be sufficient to allow sliding movement. According to U. Sachs, a clearance of 0.3mm should be present [8]. The spacing in between the fixtures is created by washers of thickness 0.42mm. The thickness of the Toray dry specimens is 0.14mm and 0.20mm for Solvay dry specimens. After consolidation both materials are of 0.13mm thickness. The polyimide tape adds 0.15mm thickness. The spacing should therefore be at least

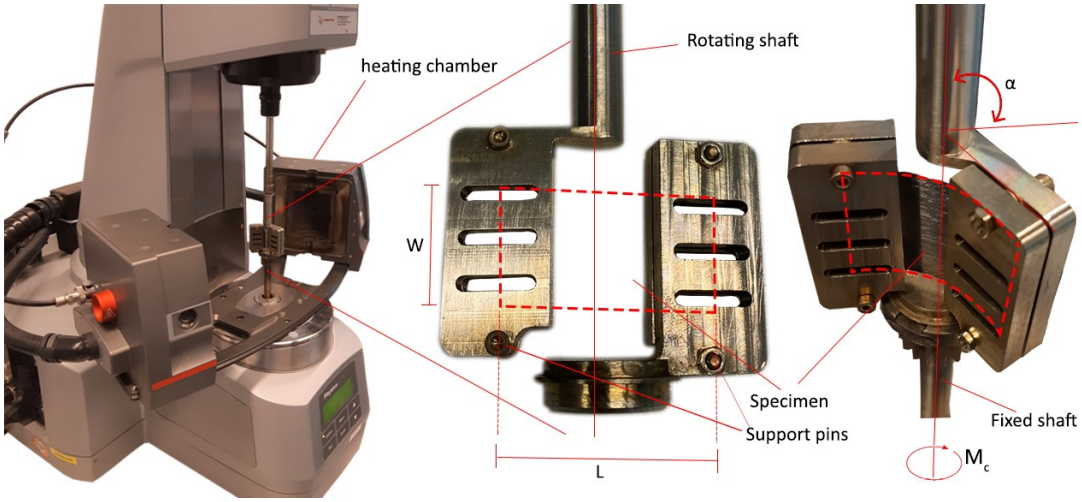


Fig. 3: Visualization of bending set-up consisting of a rheometer and oven by U. Sachs [2], edited version

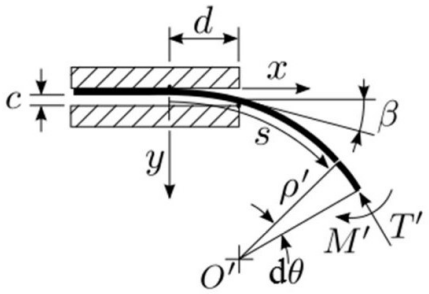


Fig. 4: Schematic representation of one fixture [8]

0.65mm, and was thus created by two washers with a combined size 0.84mm. It should be noted that the clearance varied per material, because the spacing was kept constant for all experiments.
The experiments were performed in an inert nitrogen atmosphere, which prevents degradation of the material by reacting with oxygen [2].

2.3.b Experiment parameters

The following steps were performed for each test repetition. The oven is first heated up to temperature above T_m. For Solvay to 375°C and for Toray to 365°C. The specimens are then placed in the fixture in the oven and are given a dwell time of three minutes. According to U. Sachs, three minutes is required to establish a homogeneous temperature in the specimen [2]. The specimen is then bent at set rotational velocity from 0°to 60°. All specimens are bent at 0.1, 1 and 10 rpm. A new specimen is used for every repetition. Three repetitions for each rotational velocity were performed.

2.3.c Analysis

The rheometer outputs the measured moment and deflection angle. Curvature is the conventional unit used in bending experiments. The deflection angle is therefore calculated into curvature using Equation 1.

$$\kappa = \frac{1}{\rho} = \frac{tan(\frac{\alpha}{2})}{l} \tag{1}$$

Where κ is the bend curvature, ρ the radius of the bend, α is the deflection angle and l is the arm length of the bend. The arm length is the distance between the opening of the holders of the specimen, and the shaft axis [8], this is 7.5mm.

2.4 *Microscopy*

To compare the bending behaviour of non-consolidated tapes versus consolidated tapes, the stages that both tapes go through in bending are visualized in Figure 6. In order to gain a thorough understanding of the changes the specimens go through, microscopy is performed on each stage. Non-consolidated specimens form line 1 in Figure 6, consolidated specimens form line 2.

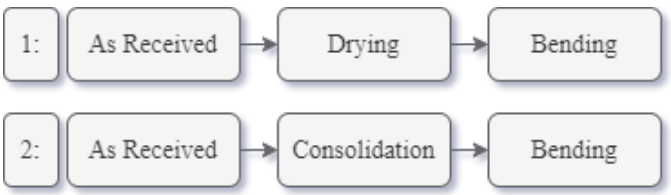
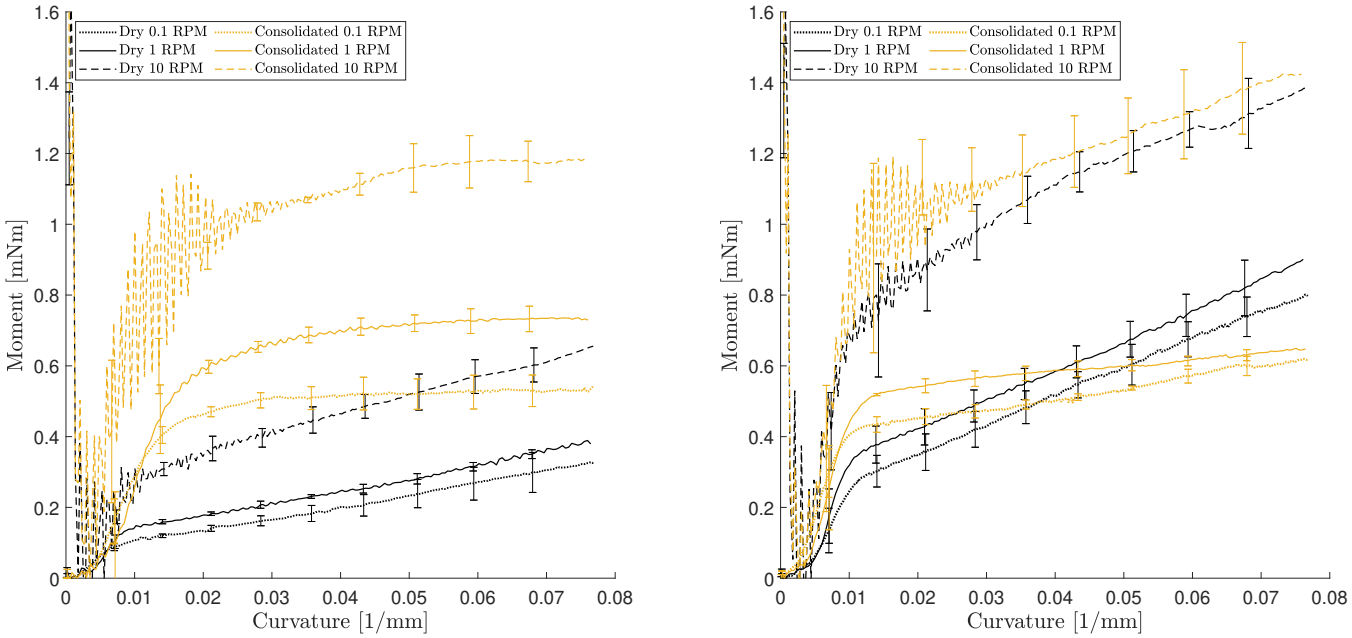


Fig. 6: Overview of stages that specimens go through during bending, which are to be visualized

Bending takes place above T_m without external pressure on the specimen, which means deconsolidation occurs. To visualize this process, micrographs of



(a) Toray

(b) Solvay

Fig. 5: Average moment versus curvature plot of bending experiments at three rotational velocities of Toray Cetex®TC1225 and Solvay APC (PEKK-FC), with $\pm$ 1 standard deviation plotted in error bars

deconsolidated specimens are also created. The deconsolidated specimens were created by carrying out the bending experiment described in Chapter 2.3.b, but terminating the cycle before bending the specimen.
As humidity dissolves into the matrix, it is not visible on the microscope. The 'dry' and 'as received' stage therefore produce a highly similar micrograph, resulting in six micrographs per material.

In order to visualize the stages using microscopy, a quarter of a specimen of each stage was cut and embedded into a cylinder of epoxy resin. After the resin had hardened, the cylinders were polished. Micrographs are taken perpendicular to the fiber direction.

3 RESULTS

The results of the bending experiments and microscopy are discussed in this chapter.

3.1 Bending

The outputs of the rheometer are processed and plotted in moment curvature graphs. For every repetition, the rheometer measured 500 data points of moment and angle. The average moment values for every curvature of three repetitions are calculated. The standard deviation of three measured moments per curvature value is calculated for every 50^{th} data point. One standard deviation is plotted above and below the average line with error bars. The results for both Toray and Solvay are displayed in Figure 5.

All measurements show a near linear behaviour after a short start-up phase. According to U. Sachs, the start-up phase can be interpreted as a viscoelastic phenomenon, where the slope is related to an elastic contribution, and the extrapolated height at zero curvature is related to a viscous contribution [2]. The measurements performed at 10 rpm show noise up until a curvature of $\simeq 0.025$ mm^{-1}. This is caused by the control system of the rheometer, which is fairly aggressive.

With increasing rotational velocity, the extrapolated height at $\kappa = 0$ increases for both materials. From 0.1 rpm to 1 rpm, the increase is relatively small compared to the increase from 1 rpm to 10 rpm. This behaviour was also found in the thesis of U. Sachs, and is further discussed in Chapter 4.

For Toray, the slopes of the dry specimens at all three rotational velocities remain relatively constant, after the start-up behaviour, compared to the consolidated specimens. The results of the consolidated specimens portray similar start-up behaviour, but take longer to become linear. After the start-up behaviour, the slopes of the consolidated specimens are all reduced, indicating a reduction in stiffness due to consolidation. Also, the slopes of consolidated specimens seems to flatten, whereas this does not occur for dry specimens.
Consolidated specimens show a higher bending moment for every rotational velocity. This indicates that consolidated specimens of Toray experience a higher viscosity. For every rotational velocity, the error bars of dry and consolidated specimens do not cross each other, meaning it is clear that consolidation results in different bending behaviour.

For Solvay, the results show a different behaviour than Toray. The results of 0.1 rpm and 1 rpm do not show an increase in bending moment due to consolidation, after a curvature of $\simeq$ 0.04 mm^{-1} curvature. Moreover, the lines of the corresponding rotational velocities of 0.1 rpm and 1 rpm cross each other. The results of 10 rpm show an increase in bending moment, but the rise is very small compared to Toray. Also, the results of 10 rpm portray no distinction in slopes. However, the slopes of the graphs of dry specimens at 0.1 rpm and 1 rpm are higher than the slopes of the consolidated specimens, which also holds for Toray.

Analysing the measured thicknesses of the specimens given in Table 2, it is found that the thickness of Solvay specimens decreases considerably due to consolidation. This is presumably caused by decrease in void content, improved fiber-matrix distribution and removal of surface roughness.

Table 2: Average thickness of test specimens with standard deviations (all results in μm)

(a) Toray

	Mean	σ
Dry	140	4
Cons.	130	3

(b) Solvay

	Mean	σ
Dry	205	11
Cons.	130	3

The average thickness of Solvay decreases by 37% where the thickness of the Toray specimens decrease by 7%. The decrease in thickness means that it may not be fair to directly compare the bending moment of dry versus consolidated.

3.2 Microscopy

Both materials are analysed by a digital microscope in the following conditions: AR, consolidated, deconsolidated and bent. The results are displayed in Tables 3 and 4. The light-grey dots portray the fibers, the grey contour around the dots denotes the polymer. Voids are visible in the micrographs as black dots and areas.

3.2.a Before bending

Multiple differences are found in the observation of the two materials before bending (rows AR and deconsolidated in Tables 3 and 4). The fiber-matrix distribution is found to be notably more homogeneous in the material of Toray. This holds for both the dry and consolidated stages. It is clearly visible that Solvay has areas where little to no fiber is present, whereas this doesn't occur in Toray.
The surface roughness of Toray and Solvay dry is found to be different. Solvay displays a clear surface roughness as its thickness varies along the specimens width. Toray does not have a similar surface roughness, even though the surface does become more rough during deconsolidation. A similar distinction between the materials is the surface wetness. The micrographs suggest that Solvay has more matrix piled up at the edges of the material than Toray.

The effect of consolidation on both materials on microscopic level is clearly visible. Consolidation improves the fiber-matrix distribution, eliminates most of the surface roughness and removes nearly all voids. Consolidation also results in a big difference in microscopic structure after deconsolidation, which is the input stage for bending. The change in microstructure due to consolidation after deconsolidation complies with the results found in the paper of T.K. Slange et al [5]. Both materials change less in microscopic structure during deconsolidation, if they have been consolidated.

The void content of both materials before and after consolidation is found to be very similar. However, Solvay seemed to be more prone to creating voids, especially during deconsolidation of dry material.

Table 3: Micrographs of Toray Cetex®TC1225

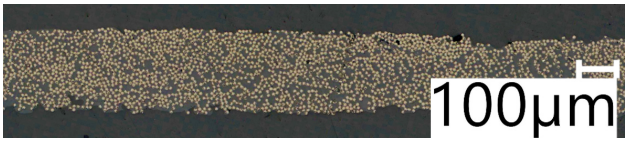
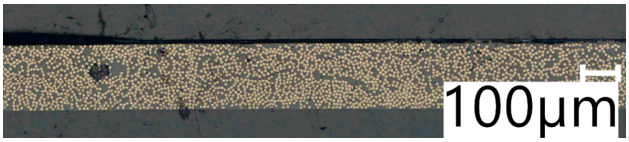
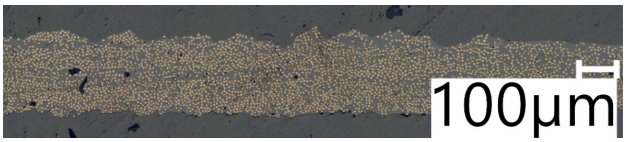
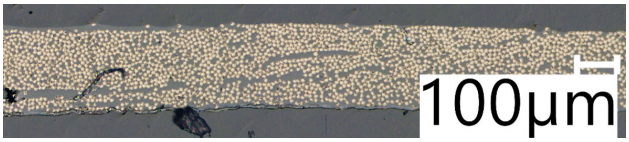
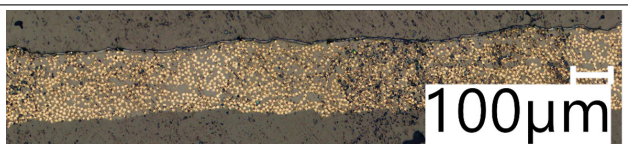
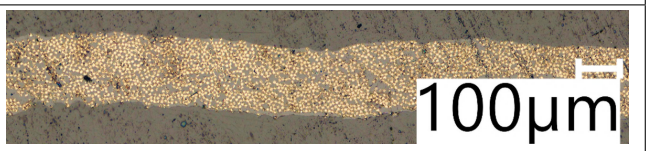
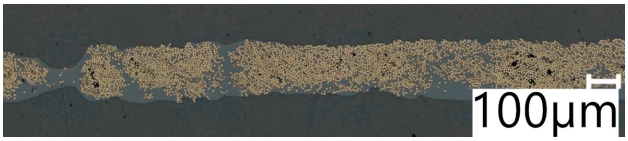
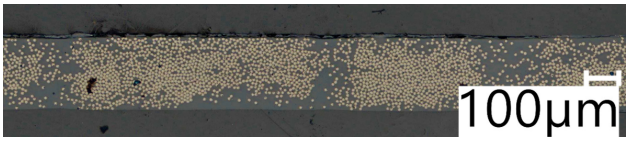
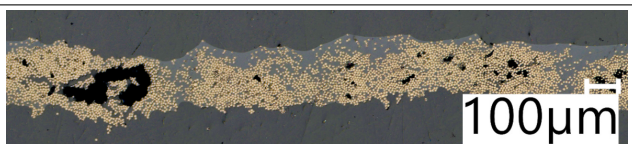
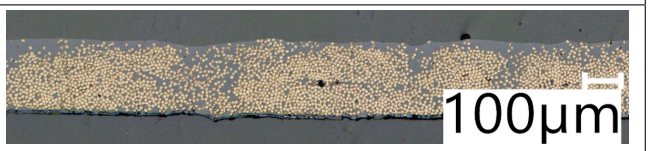
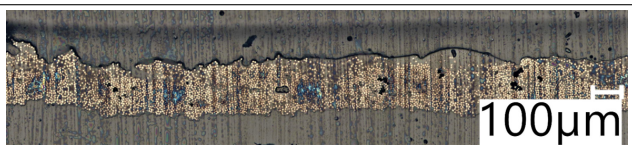
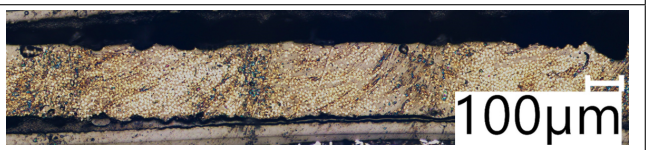
	Dry	*Consolidated*
AR		
Decons.		
Bent		

Table 4: Micrographs of Solvay APC (PEKK-FC)

	Dry	*Consolidated*
AR		
Decons.		
Bent		

3.2.b After bending

The micrographs of the bent specimens are of worse quality. The process of polishing the epoxy cylinders was flawed, causing scratches and visible bubbles of air in the epoxy. The graphs are, however, of sufficient quality to be observed and analysed. The consolidated Solvay specimen portrays big black areas around the specimen. This is assumed to be the shadow of the specimen deeper into the epoxy cylinder.

The micro-structure of the bent specimen of consolidated Toray looks neater and still more compact than the dry specimen. Also, the dry specimen has some surface roughness, which is not present on the consolidated specimen. No difference is found in void content. It is clear to conclude from the difference in micrographs of Solvay dry and consolidated that consolidation heavily influences the micro-structure during bending. The dry specimen has visible voids, an irregular surface and worse fiber-matrix distribution than the consolidated specimen.

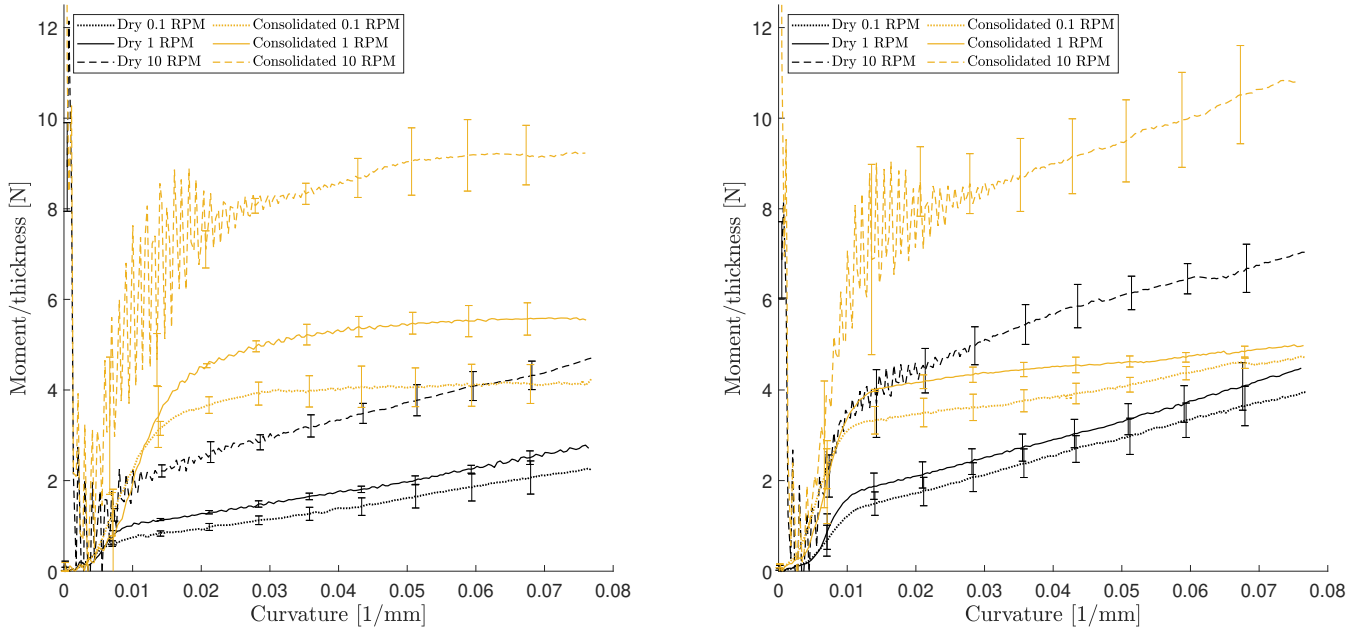
4 DISCUSSION

4.1 Bending behaviour

In Chapter 3.1, it was noted that the thickness of consolidated specimens of Solvay decreased by 37%. Thickness should affect the measured moment, according to the Bernoulli-Euler beam theory of Equation 2.

$$M = EI\kappa \tag{2}$$

Where I is the area moment of inertia and E the Young's Modulus. The Bernoulli-Euler theory is



(a) Toray

(b) Solvay

Fig. 7: Average moment/thickness versus curvature plot of bending experiments at three rotational velocities of Toray Cetex®TC1225 and Solvay APC (PEKK-FC), with ± 1 standard deviation plotted in error bars

based on isotropic material. Fiber reinforced thermoplastic composite is not isotropic, but this theory is commonly used to approximate the bending moment. The value of I is relatively unknown for thermoplastic composite, due to the complexity of the material. It is however certain that the thickness does affect this value. To eliminate the influence of the variation in thickness, the moments measured in the experiments were divided by the thickness. Figures 7a and 7b portray results compensated for the change in thickness.

The results in Figure 7 show a clear increase in bending moment/thickness of consolidated specimens compared to dry specimens for both materials. The error bars do not overlap for any rotational velocity. The results again portray lower stiffness for consolidated specimens, and a higher viscosity. Whether this is a valid way of processing the data is unknown, because there is not enough knowledge of fiber reinforced thermoplastic material.

The measured moments in Figure 5 increase with increasing rotational velocity. The Young's Modulus of the carbon fiber itself is constant, meaning that the area moment of inertia (I) cannot be assumed to be constant. According to U. Sachs, interlocking of fibers by entanglement is likely to increase at higher deformation rates [2], causing more intra-ply shear. This could justify the relatively small increase in moment from 0.1 rpm to 1 rpm, and the large increase from 1 rpm to 10 rpm.

4.2 Influence of micro-structure

Research of U. Sachs concludes that thickness of multi-layer laminates (created by adding more layers) does not effect the Young's Modulus [2]. However, the influence of varying the thickness of a single-layer prepreg is unknown. Sachs also states that intra-ply shear is influenced by change in lay-up of the composite [2], suggesting that a change in micro-structure due to consolidation, as observed in Chapter 3.2, can effect the bending behaviour of the material. To support this argument, Sachs also concludes that the bending deformation of UD laminates is dominated by intra-ply shear deformations and fiber bending [2]. The micrographs of Chapter 3.2 and the measurements of the thicknesses of the specimens show that consolidation improves fiber-matrix distribution, removes surface roughness, reduces voids and results in a more compact structure. This therefore suggests that the more compact micro-structure of

consolidated materials increases in-plane shear in a single-layer prepreg, resulting in a higher viscosity and lower stiffness.

A research into measurement of the influence of varying thickness of a single-layer UD fiber reinforced thermoplastic tape on bending and in-plane shear is advised. This aids in creating more accurate material models of this type of material, and in validating whether eliminating the effect of thickness on bending moment is a well-founded way of processing the results of this research.

T.K. Slange et al. suggest that the driving force for deconsolidation is frozen-in-fiber stresses. Fiber stresses are relieved during press consolidation [5]. Micrographs of press-consolidated-deconsolidated and as received-deconsolidated of research of Slange et al. show very similar results as this research. The fiber stresses are relieved by allowing deformation of the fibers into a lower stress state [5]. This validates the microscopy results found in this research. Although not further researched in the scope of this work, it is suggested that the fiber stresses still present in dry specimen during bending, may cause an effect on the stiffness of the material. Further research into the change in stiffness due to consolidation is required.

4.3 Further recommendations

The bending and microscopy experiments were performed in a matter of a few weeks. This meant that there was only time to create micrographs of one specimen representing one stage, and to do three repetitions of each bending experiment. There are many possible small variances in the material that can have an effect on the bending behaviour. Besides variance in thickness, there is also variance in fiber fraction, fiber stresses and other material properties. To gain more reliable results, it is recommended to do a more extensive research where more repetitions of the experiments are performed.

In order to fully characterize the bending deformation mechanism, the start up behaviour up until a curvature of $\simeq$ 0.02 curvature needs to be researched. The reason for the high stiffness in the start up phase is unknown. The reason for the longer start-up behaviour for consolidated specimens is also unknown.

Ultimately, it is desirable to quantify the bending behaviour of consolidated specimens and test those values in stamp forming simulations. By comparing the wrinkling predictions to real-world produced parts, the improvements to the simulations can be validated.

According to T.K. Slange et al., the thermal expansion of moisture causes deconsolidation [5], which is a cause for voids. Voids were found in micrographs of Solvay dry. The influence of voids on bending behaviour is unknown. An investigation into the influence of humidity and voids on bending behaviour is therefore required.

The experiments in this paper were carried out at 365°C for Toray and at 375°C for Solvay. However, experiments by U. Sachs conclude that bending behaviour is also dependent on temperature, given the same thickness and rotational velocity [2]. In order to fully characterize the material, it is necessary to research the influence of temperature on bending behaviour.

5 CONCLUSION

This paper presents results of bending experiments, that research the influence of consolidation on bending behaviour of single-layer UD fiber reinforced thermoplastic tapes in molten condition. Two materials were used to confirm the reliability of the performed experiments; Toray Cetex ®1225 and Solvay APC. Micrographs of specimens that have gone through relevant stages of bending at temperature above T_m are also presented. It can be concluded that consolidation does influence bending behaviour. The consolidated specimens show a clear increase in bending moment compared to non-consolidated specimens, but a reduction in bending/curvature slope after similar start-up behaviour. The lower slope indicates a reduction in stiffness for consolidated specimens. The increase in bending moment is caused by an increase in in-plane shear, resulting in higher viscosity. Micrographs presented in this paper, combined with results from previous researches, suggest that the change in bending behaviour is caused by a difference in micro-structure due to consolidation. Consolidation improves the fiber-

matrix distribution, eliminates most of the surface roughness and removes nearly all voids. Also, press-consolidation relieves frozen-in-fiber stresses, which influences the micro-structure of the material after deconsolidation. This results in a more compact micro-structure, which causes an increase in in-plane shear. In-plane shear, together with pure bending, are the two dominant factors in bending of thermoplastic composite [2].

Currently, material models of the bending deformation mechanism used in simulations of stamp forming this material are based on characterization of non-consolidated specimens. However, consolidated laminates are the input of stamp forming. It is advised to review this decision, knowing the change in bending behaviour of a single-layer UD fiber reinforced thermoplastic due to consolidation. Material characterization should be performed on consolidated single-layer UD fiber reinforced thermoplastic tapes, and tested in simulations of stamp forming. These simulations need to be compared to real world stamp formed parts to confirm better accuracy of results of the simulations.

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