INVESTIGATION OF TESTING MECHANISMS BY DOPING OF BONDING SURFACES WITH NANO PARTICLES

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ABSTRACT

Fiber bridging in front of the crack tip was observed during the wedge peeling of in situ consolidated thermoplastic samples. Therefore, it was investigated, whether the measurements still represent the bonding strength of the welding. For identification the bonding area needs to be made detectable. Therefore, nano particles were dispersed in a solvent, which then was brushed on the tape surfaces. From these doped tapes samples were in situ consolidated, peeled, and examined by SEM. It was found that the fracture is often several micrometer or fiber diameters beneath the bonding plane. This behavior is not a doping effect, but a result of the formation of bonding zone during processing.

The wedge peel resistance measurement consequently does not characterize only the matrix bonding in the welding zone, but it characterizes the bulk material performance after in situ consolidation.

1 INTRODUCTION

At different industrial ranges large interest exists in the introduction and/or further spread of continuous fiber reinforced thermoplastics for complex shaped parts [1, 2]. Besides the well known autoclave technology, commonly used for producing thermoset based parts but also applicable for manufacturing thermoplast based parts, which is, however, only economical for small-scale production or prototype development, two other technologies are available for large-scale production. The first is the organic sheet processing chain, consisting of manufacturing fully consolidated organic sheets, to thermoform them, and, if necessary, welding them [3]. The second possibility is to use fully impregnated continuous fiber reinforced thermoplastic tapes [4, 5]. These tapes can be used in a winding process [6] or, to create small up to large as well as complex shaped parts, in a tape placement process [7, 8]. The high quality requirement of the components require adequate production processes and a deep understanding for the parameters affecting the process [9, 10]. Compared to a thermoset based process the thermoplastic tape placement offers the possibility to save an additional consolidation treatment due to the in situ consolidation. During the in situ consolidation a net-shape placement of the material and at the same time welding of the substrate in one step takes place.

At the Institute für Verbundwerkstoffe (IVW) intense work was performed in the field of thermoplastic tape placement with in situ consolidation [11]. The evaluation of the resulting material performance is carried out by comparison of the tape placement and the autoclave process by performing tests, e.g. short beam bending, on samples from

multi-layer plates manufactured. For the process optimization only two tapes are welded and investigated by a wedge peeling test.

It is well known and e.g. Johnson and Mangalgiri [12] and Bradley and Cohen [13] described for thermoset composites, that the fracture is leaving the bonding plane if a good bond is getting stronger than the defects in the surrounding material. This can be explained by the stress field in front of the crack tip acting in adjacent layers as well. If a defect makes the adjacent plane weaker than the bonding plane, the fracture switches and creates fiber bridging (Fig. 1). This means that some fibers, which were on the upper side of the crack, do bridge in front of the crack tip and becomes part of the lower side. While the crack is continuing to grow, the bridging fibers are pulled out and/or break. The pulling out and the breaking is then part of the measured value as well as the crack growth in a layer which is not the bonding layer anymore.

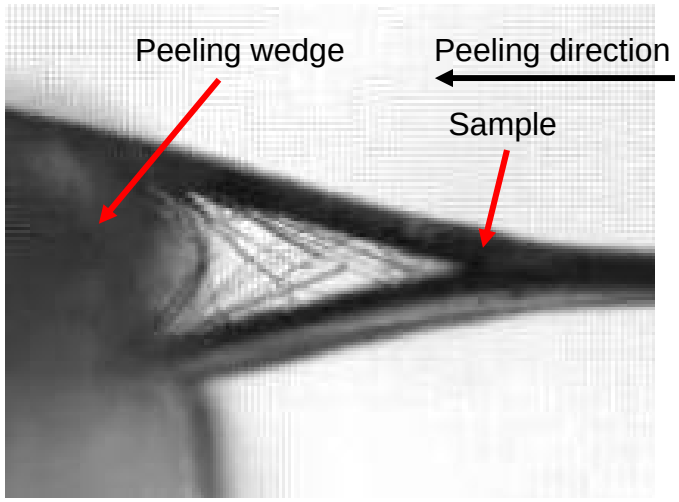

Fig. 1: Multiple fiber bridging

2 EXPERIMENTAL DETAILS

2.1 Materials

Typically carbon fiber reinforcements and high performance thermoplastic materials like PEEK or PPS are used for thermoplastic tape placement processes. In this study a CF/PEEK tape material manufactured by SUPREM was used. The tape was of rectangular shape having a width of 12.0 mm and a thickness of 0.15 mm. The fiber volume content was adjusted to 55 %.

Spherical shaped nano particles of different size and material were used. As the first material, TiO2 particles with a diameter range of 200-300 nm (type: 2310, Kronos) and a diameter of 15 nm (type: RM 300, Sachtleben) were selected. The second material was ZnS and the particles' diameter was 300 nm (type: HDS, Sacholit).

2.2 Procedures

The nano particles were dispersed in isopropanol up to a particle volume content resulting in a viscosity of the mixture allowing brushing the dispersion on the tape's surface. Different mixtures were used to identify the most homogenous distribution of particles on the surface. After evaporation, small agglomerates of the particles were found ho-

mogenously distributed on the material's surface. The smaller sized TiO2 particles exhibit best performance.

Some further tests were performed to analyze whether the particles adhere to the surface while processing the tapes. Already the not further treated specimens show good adhesion. A preheating of the doped tape surface using IR-heaters result in covering of the particles by molten matrix material, but the heating also results in a changed surface roughness of the tape material. So, it was decided to use the doped tapes without any further treatment.

The in situ consolidation was run on a special test rig allowing processing the tape material in a tape placement configuration, i.e. consolidation between a flat tool and a consolidation roller. Standard processing parameters were adjusted to:

- Tool temperature: 265 °C
- Roller temperature: 95 °C
- Consolidation pressure (applied on pressure cylinder): 4.6 bar
- Placement velocity: 3 m/min
- Main heater (hot gas torch): variable

For further characterization the resulting in situ consolidated tape pairs were investigated using scanning electron microscope and wedge peel resistant testing. Wedge peel resistance was selected since it can be used to characterize the welding of two thin tapes [14] and, although the loading mode is different, it can be used for process optimization due to the fact that the maximum properties in regard to the process parameters are identical for both wedge peel resistance tests and standard tests like e.g. short beam bending or double cantilever beam test [15].

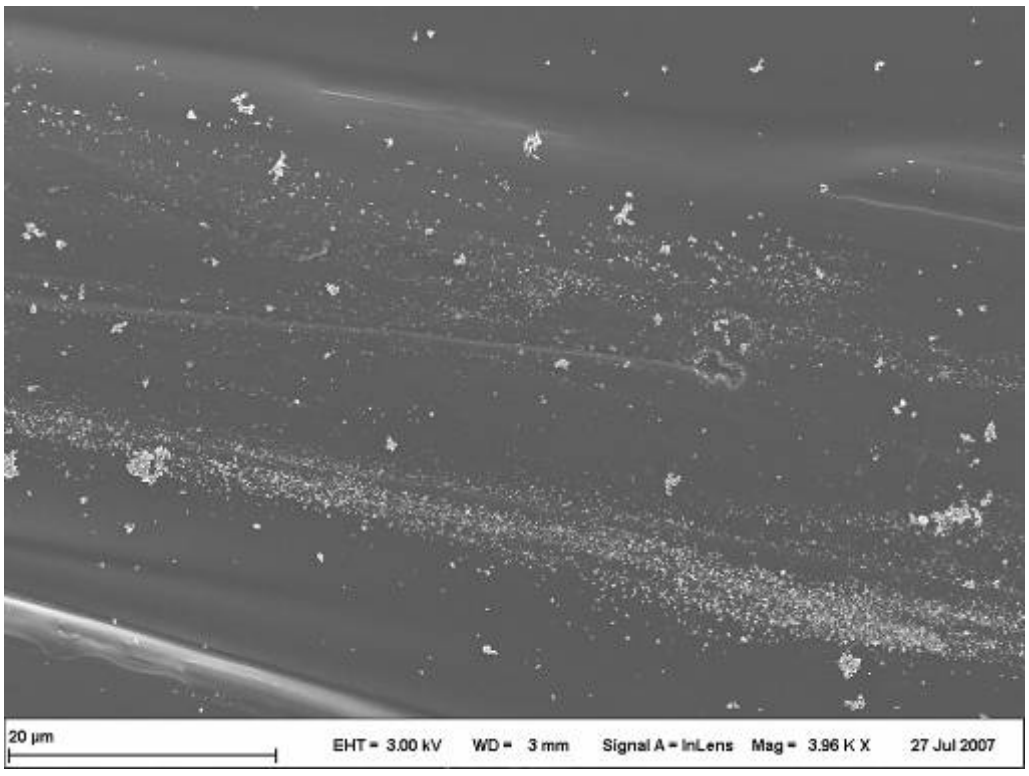

Fig. 2: Tape surface doped with TiO2 nano particles

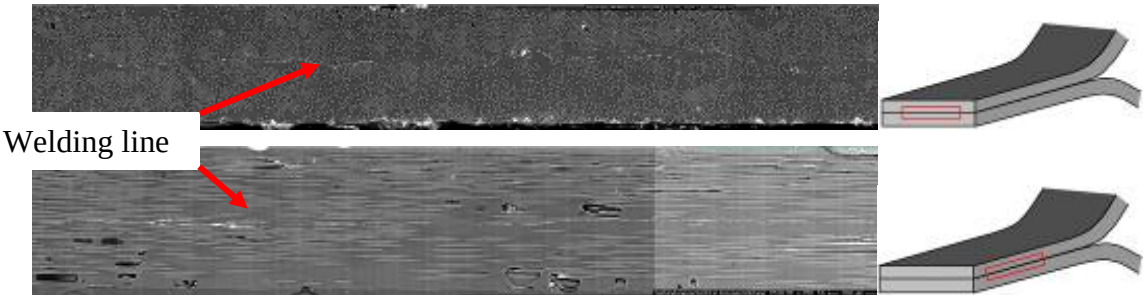

Fig. 3: Cross section after welding perpendicular (upper one) and parallel (lower one) to welding direction

3 RESULTS

The aim of surface doping using nano particles was to mark the tape surface allowing identifying the original tape surface after in situ consolidation process. From the screening on different nano sized materials a TiO2 was selected due to a good spreading all over the tape surface (Fig. 2). After in situ consolidation cross sections, both perpendicular and parallel to the process direction, were examined to identify the welding line (Fig. 3). Due to the difference in material the TiO2 result in good contrast and the welding line can be detected very well. Already at this stage it can be seen from the perpendicular cross section, the deformation after in situ consolidation of the individual tape differs from point to point. Whilst the overall thickness of the welded tape pair is almost constant, the weld line indicated by the nano particles is not located in the mid all over the tape width (Fig. 4). Obviously, pronounced material flow might occur during the in situ consolidation, which is also indicated by the dispersed location of the nano particles (Fig. 4, mark “I”) used for tape surface indication.

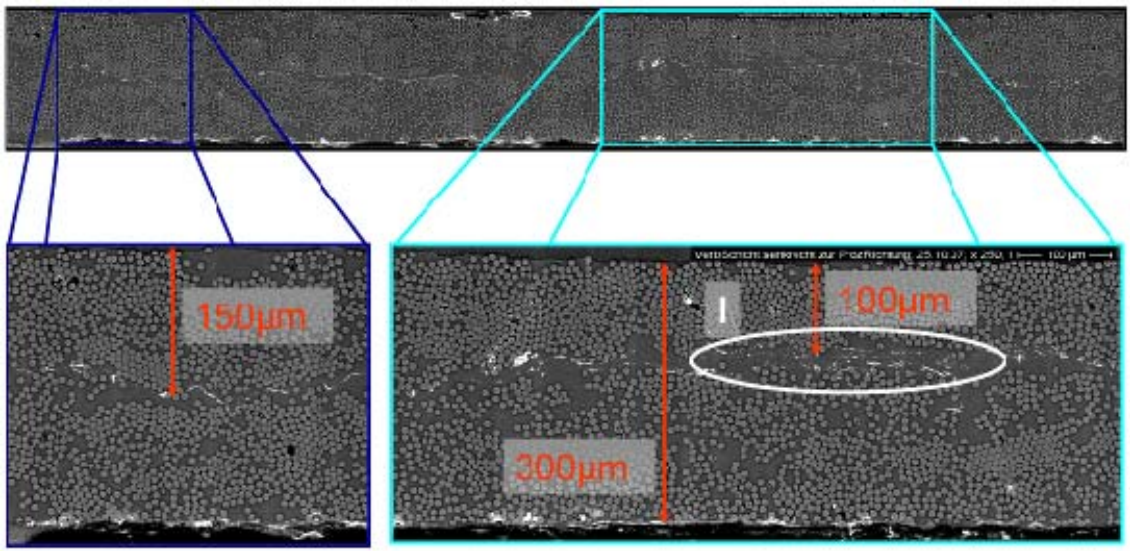

Fig. 4: Tape deformation due to in situ consolidation

If performing wedge peel testing on different samples manufactured with the same parameters, the resulting peel force is on an almost constant level exhibiting a more or less pronounced scattering for each specimen. And, the average value of peel force ranges on the same level for all specimens (Fig. 5). Analyzing the peel force curves more detailed, it can be seen, there are several peaks with significantly reduced peel force.

These peaks can be correlated with visible stripes on the peeled tape surfaces (Fig. 6). Investigating the different areas of the peeled surface, a more ductile matrix failure is found outside the stripes whereas in the stripes' region fiber failure and fiber matrix interface failure is found.

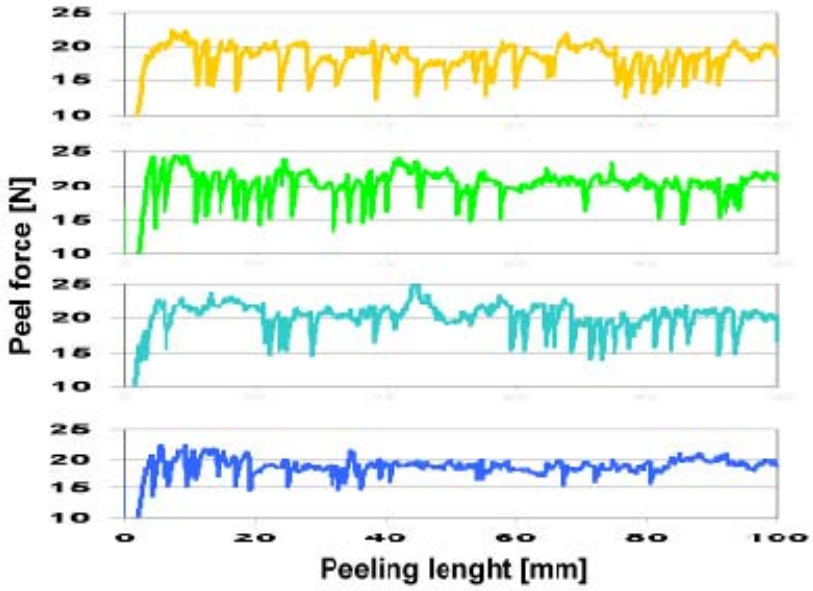

Fig. 5: Peel force vs. peeling length for different specimens manufactured with same parameters

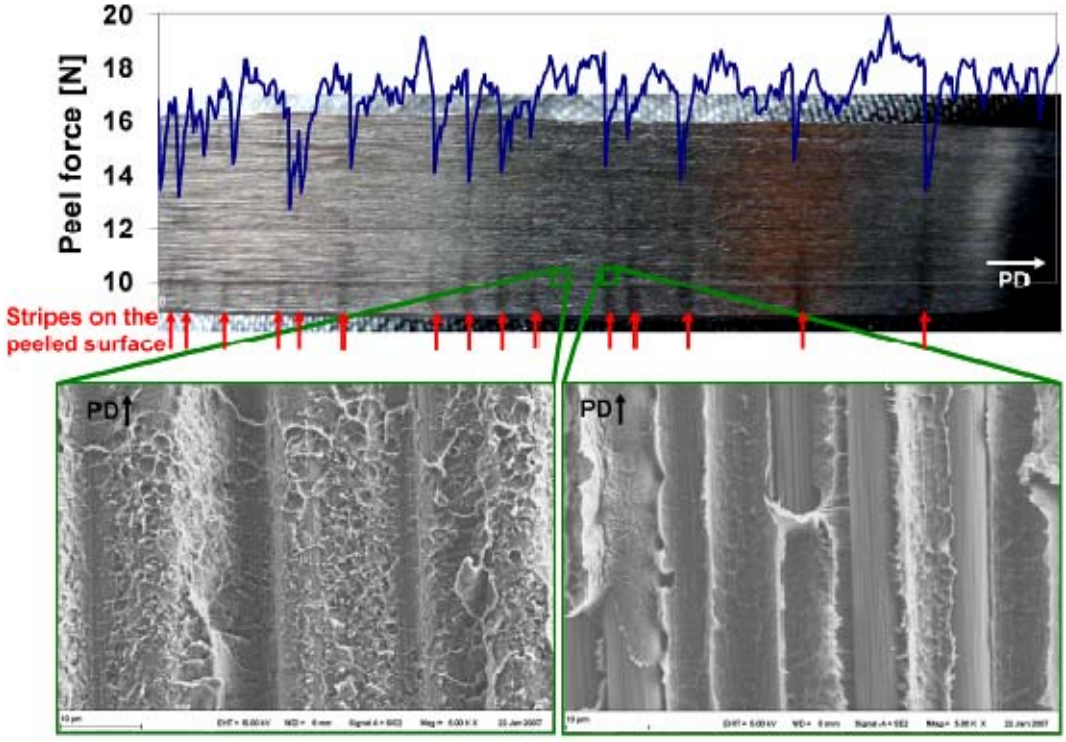

Fig. 6: Correlation between peaks in peel force curves and stripes on peeled tape surfaces (top) and peel surface analysis in the stripe region (lower right) and outside (lower left) – "PD" indicates the peeling direction

Analyzing the cross section perpendicular to the peeled surface, areas in which the doped tape surface is located below the peeled surface can be found (Fig. 7). This demonstrates the peeled surface does not completely represent the welding zone.

To verify whether the nano particle doping affects the peel resistance or not, a comparison with varying gas volume flow was done. It was found, the wedge peel resistance of doped specimens ranges almost in the scattering range of undoped specimens (Fig. 8). So, no pronounced influence of nano particle doping on the tape surface was detected, as it is found for bulk material doping [16].

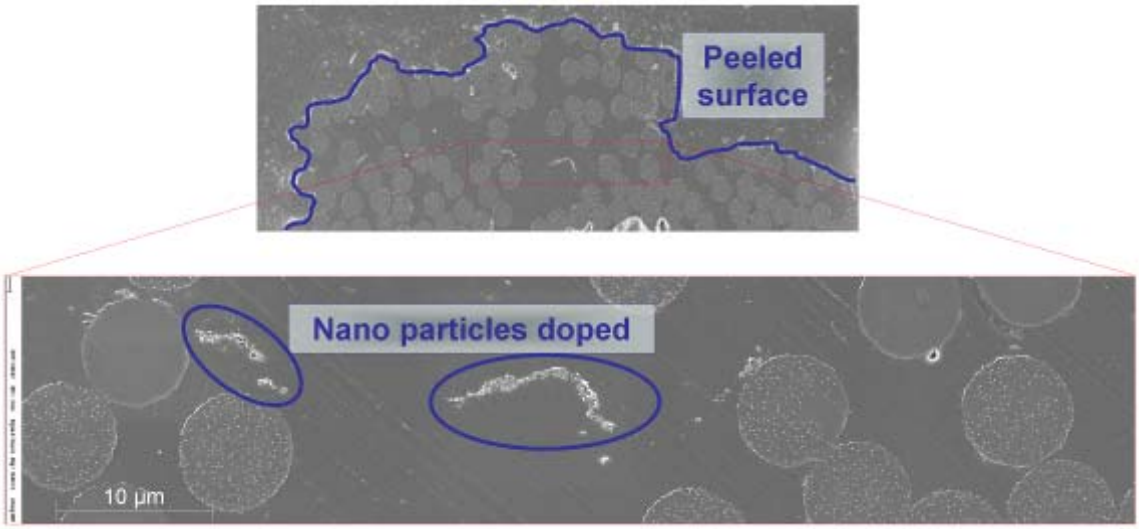

Fig. 7: Cross section perpendicular to peel plane showing the doped tape surface clearly below the peeled surface

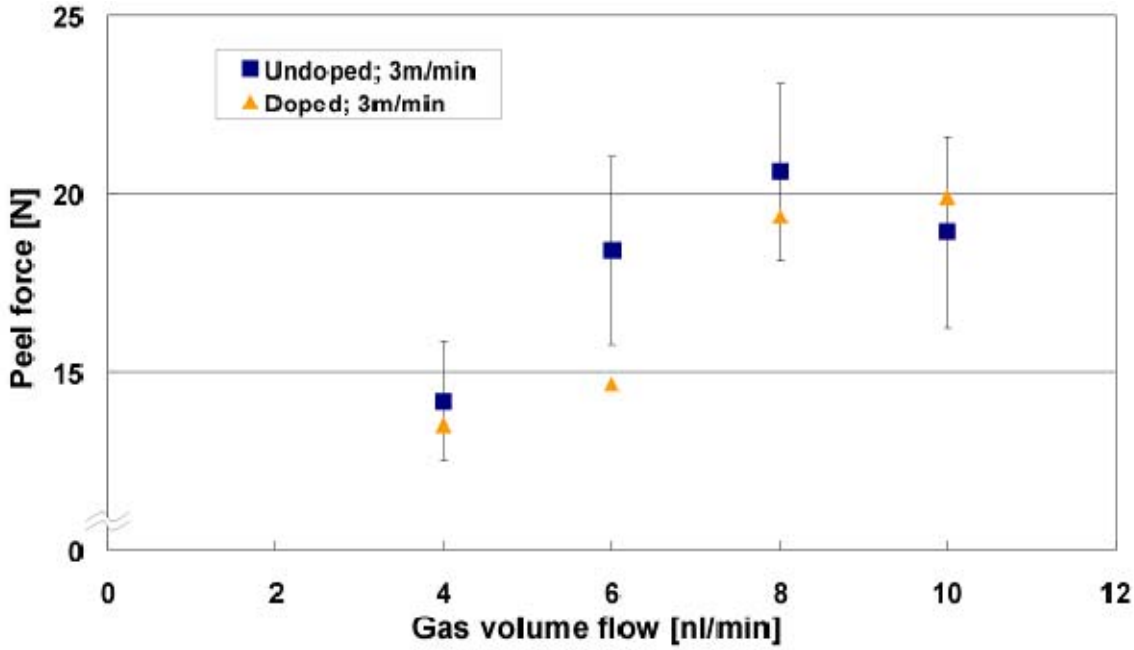

Fig. 8: Peel force vs. gas volume flow during placement process for doped and undoped specimens

4 DISCUSSION

Assuming a homogeneous tape material, good fiber matrix adhesion, and in situ consolidation resulting in matrix performance in the welding zone comparable to bulk material behavior one could expect to receive a plain peeling surface during testing. Already prepreg based hand laminated and autoclave processed specimens, which might be most comparable with before mentioned best case scenario show fiber bridging effects and the fracture surface does not represent the former prepreg surface anymore. Reasons for this behavior are mainly due to imperfections already in the prepregs used. Prepreg surfaces exhibit a more or less pronounced surfaces roughness, the filaments are not homogeneously distributed over the prepreg cross section, and they are not well elongated. Furthermore, voids in the matrix might exist or filaments are not fully impregnated.

During the in situ consolidation the tape material is deformed due to the consolidation pressure. The resulting welding line exhibits a more complex structure compared to the original tape surface and due to flow processes single filaments or filament bundles of one tape penetrate the cross sectional area of the second tape (Fig. 9). The forward moving peel crack tip tends to follow the embedded filaments until the resulting fracture surface will need more energy to be generated than fiber cracking will need. Only in case of a significant lower bonding strength in the welding zone the peel crack tip will follow the contour of the welding zone.

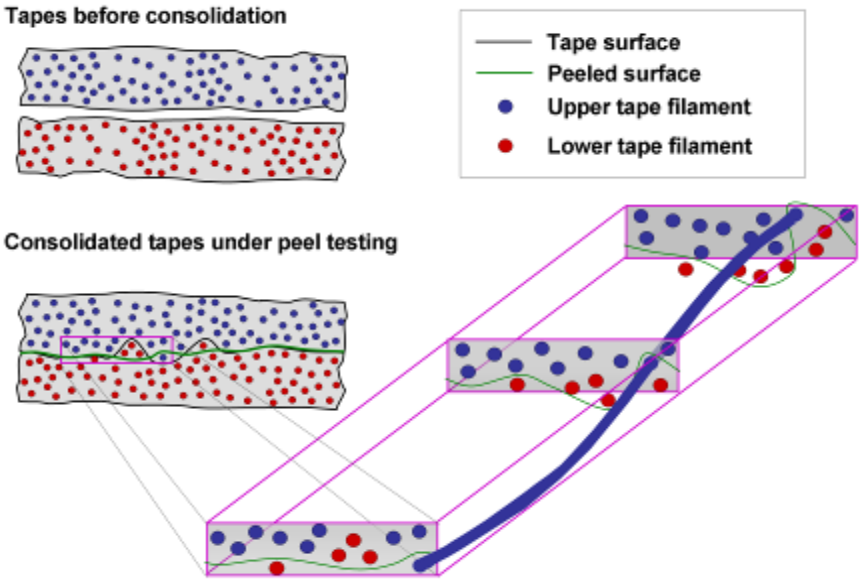

Fig. 9: Peel crack tip development

In fiber rich regions the tape deformation due to consolidation pressure (often in combination with inappropriate processing conditions) can generate cracks significantly weakening the material (Fig. 10). Such materials will generate a peeling surface defined mainly by pre-existing damages. So, the welding zone, whether weak or not, will not contribute the peeling.

Summarizing it can be stated, the peeling surface will not necessarily represent the welding zone in both cases, whether for good bonding nor for less optimized bonding.

So, the question is coming up, if the peel testing is a method to characterize the in situ consolidation or not. Since the in situ consolidation is aimed to be a final processing step, the resulting properties of the whole material are of interest. Whether the bonding in the welding zone is the weakest point or other defects weaken the material characteristics is of minor interest. From the testing the sensitiveness of wedge peel resistance regarding different process parameters during in situ consolidation is verified and correlates with test results gained on bulk samples, e.g. short beam shear testing or double cantilever beam testing [14, 15]. The crack formation mechanisms found for wedge peel testing as described before are also found for bulk samples.

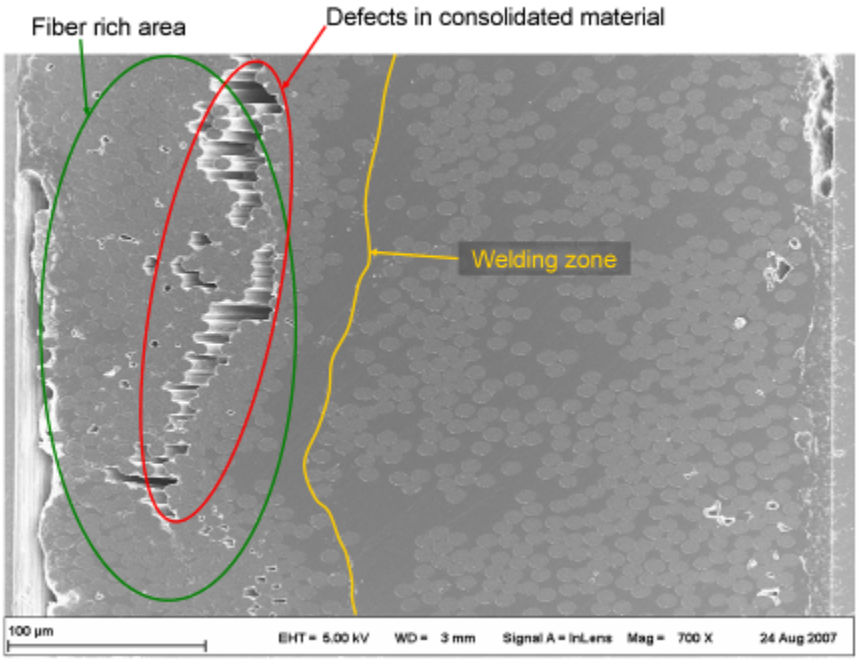

Fig. 10: Damages in consolidated material due to pre-existing damages in the tape material used and inappropriate processing conditions

5 CONCLUSIONS

For process optimization a fast and easy to perform testing method is required. Wedge peel resistance testing allows studying the effect of individual process parameters regarding resulting bulk material performance, although the loading mode during testing as well as loading conditions in front of the crack tip are not well defined. Consequently, the resulting wedge peeling force can not be used for quantifying the energy release rate. But, wedge peel testing was used for process optimization and as a result the in situ consolidation process was optimized to reach interlaminar shear strength values directly comparable with autoclave process [17].

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