

# Short time degradation of thermoplastic composites during stamp forming

#### Internship report

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### Summary

The use of thermoplastic composites has increased rapidly in the automotive and aerospace industry in the last years. Carbon fiber reinforced polyether-ether-ketone (C/PEEK) is a high performance thermoplastic composite which is suitable for different components and production techniques, one of those techniques is stamp forming. During the stamp forming process the thermoplastic composite laminate needs to be reheated above melt, which can induce degradation. Therefore the objective of this research is to use realistic heating settings to determine short time degradation during the stamp forming process. 8-ply laminate are heated using an infra red (IR) oven set to temperatures of 445°C (for 95, 240 and 480 seconds) and 480°C (for 55 and 150 seconds) before being formed. Differential Scanning Calorimetry (DSC) measurements are used to determine the crystallinity content of the treated laminates, no significant changes are observed in the results. This is confirmed by 3-point bending measurements where no significant decrease in mechanical performance is observed. Therefore this research shows that the chosen heating parameters do not result in degradation of 8-ply C/PEEK laminates.

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### Abbreviations

DSC	Differential Scanning Calorimetry
IR	Infra Red
(C/)PEEK	(Carbon) Polyether ether ketone
PI	Polyimide
TPRC	ThermoPlastic composites Research Center
UD	Unidirectional

### **1** Introduction

#### 1.1 Background

Composites are more and more common in nowadays industry. Especially within the automotive and aerospace industry weight reduction and fuel economy are becoming more important and drive the industries in using advancements in material use. Two of the most recent examples in the aerospace industry are the Boeing 787 [1] and the Airbus A350 [2], where at least 50% of the materials used are composites. These composites consist of a polymer matrix which is reinforced with continuous fibers. The polymer matrix can either be a thermoset, for example epoxy, phenolics or polyester, or a thermoplastic polymer, for example PEEK, PEI, PP or PPS. These polymer matrices are often reinforced with carbon, E- or S-glass or aramid fibers.

#### Degradation

Thermal degradation is an irreversible chemical reaction within the PEEK polymer which causes a loss in material properties. PEEK starts to degrade at a temperature of 380°C and several researches are done on this subject. Previous research on C/PEEK laminates at TPRC was done at temperatures from 385 to 445°C in an air convection oven with hold times of 10, 30, 60 and 90 minutes. DSC measurements showed a decrease in crystallinity to only 5% for a heat treated specimen at 445°C for 90 minutes [3, 4]. Kim et al. [5] performed short time heat treatments with temperatures varying from 540 to 640°C for 8 to 60 seconds. This was done in a nitrogen environment with ceramic heat radiation.

#### Stamp forming

The use of fiber reinforced thermoplastics has increased over the last few years due to advances in production techniques and the ability of a thermoplastic to be reheated above its melting temperature and reshape the material. In general process conditions for high performance thermoplastics are in the range of 300 to 400°C and 1 to 20 bar [6]. An example of a production technique is press-forming. During this process a laminate is heated above melt temperature and then formed into the desired shape, as visualized in figure 1.1. Quite some research has been done on the stamp forming process



Figure 1.1: Stamp forming process, reproduced from ETH Zurich [7].

with respect to the change of fiber orientation, formability and final product performance, for instance by Haanappel [8].

#### 1.2 Objective

Although quite some research into degradation and stamp forming has been performed, little is known about possible degradation during the stamp forming process. Therefore the objective of this research is to use realistic heating settings, IR oven temperatures and heating times, to determine short time degradation during the stamp forming process.



This section contains the approach of the research. First the material which is used is introduced and the manufacturing process is discussed. This is followed by the heat treatment conditions and procedure and the test methods which are used to determine whether degradation occurred. Differences between the first test set and the second test set are described separately.

#### 2.1 Material

The material used for the experiments is a carbon PEEK resin system manufactured by TenCate Advanced Composites, otherwise known by its trade name: TenCate Cetex<sup>®</sup> TC1200 PEEK Resin System [9], the most important properties are presented in table 1. The material is provided as a UD tape with a width of 12 inch (30.48 cm).

Property	Orientation	Result	Unit
Tensile strength	0°	2280	MPa
Tensile strength	$90^{\circ}$	86	MPa
Flexural strength	90°	152	MPa
Melt temperature	-	343	°C
Typical processing temperature	-	370 - 400	°C

Table 1: Important properties TC1200 resin system [9].

#### 2.2 Laminate manufacturing

Multiple laminates are produced for the heat treatments. However the manufacturing procedure for the first test set was slightly different than for the second test set. The laminate layup and consolidation cycle however are identical for both manufacturing procedures.



**Figure 2.1:** Schematic of the press, a PI film is placed between the caul sheets and the laminate.

**Figure 2.2:** Thermocouple layout and sample cuts (along dashed lines) for the first test set. Thermocouples are displayed as arrows.

#### First test set

For the first test set a large sheet of  $600 \text{ mm} \times 600 \text{ mm}$  was produced with embedded thermocouples, a schematic of the press build up is displayed in figure 2.1. The position of the thermocouples and the layout of how the samples for the heat treatment were cut is displayed in figure 2.2, the thermocouples have been placed on the symmetry line and one layer under the top.

#### Second test set

The second test set was produced in a 12 inch picture frame, a section view of the laminates in the picture frame is displayed in figure 2.3, again PI films are placed between de caul sheets and the stacked plies. The picture frame method was used to reduce the amount of fiber flow out during the consolidation cycle.



Figure 2.3: Sectional view of picture frame with the two ply stacks.

#### Layup

The layup which is used for this research is a  $[0_3/90]_s$  layup, as displayed in figure 2.4. This layup is chosen for the 3-point bending test to have the most stress in transverse layers and therefore a polymer dominated fracture behaviour. The same layup has also been used by Kumar [4], which makes it more easy to compare the results.



Figure 2.4: Schematic of layup, reproduced from [4].

#### Consolidation cycle

The laminates are consolidated with a TPRC standard cycle for the TC1200 resin system, which is displayed in figure 2.5. The press is heated to  $385^{\circ}$ C with a rate of  $10^{\circ}$ C/min under a pressure of 2 bar. After 10 minutes at  $385^{\circ}$ C the pressure is increased to 20 bar and kept at these settings for another 20 minutes, after which the laminate is cooled to  $120^{\circ}$ C with a rate of  $-5^{\circ}$ C/min and to  $60^{\circ}$ C with a rate of  $-10^{\circ}$ C.



Figure 2.5: Consolidation cycle set point. With the temperature in blue and the pressure in red.

#### 2.3 Stamp forming (heat treatment)

The samples obtained from the composite sheet were heat treated using IR heating, as used in the stamp forming process. The heat treatments were varied by changing different process settings: IR oven temperature and heating time. For the first test set 3 laminates per test condition were used to measure possible differences within a test condition, this has not been repeated for the second test set. During the test process laminate specimens were placed on a PI film which was taped to a carrier frame, as displayed in figure 2.6.



Figure 2.6: Test setup for stamp forming.

#### First test set

For the first test set the IR oven temperature and laminate temperature were predetermined. The heating times however were determined with an additional piece of laminate. This was done because

the press can not control the stamp forming process with the help of the added thermocouples. An overview of the test conditions is given in table 2.

Condition	1	2	3
Oven temperature [°C]	445	480	480
Time [s]	95	55	150
Laminate temperature [°C]	385	385	435

#### Table 2: Heat treatment conditions first set.

#### Second test set

For the second test set the IR oven temperature was set at 445°C and the heating times were fixed at 95 seconds, 240 seconds and 480 seconds. With these settings the first condition of this second test set is the same as the first condition of the first test set. The temperature during the complete cycle was monitored with a thermocouple. Again an overview of the test conditions is given in table 3.

 Table 3: Heat treatment conditions second set.

Condition	1	2	3
Oven temperature [°C]	445	445	445
Time [s]	95	240	480
Reached laminate temperature [°C]	360	385	390

#### 2.4 DSC

After stamp forming the first test set, several specimens were tested with the differential scanning calorimetry apparatus (DSC). The specimens were tested with a single cycle method, because the crystallisation behaviour was of importance for this research and a double cycle only ensures representable melting behaviour. The method consists of three phases:

Phase 1: The specimen is heated from  $50^{\circ}$ C to  $400^{\circ}$ C with a rate of  $10^{\circ}$ C/min.

Phase 2: The specimen is kept at 400°C for 10 minutes.

Phase 3: The specimen is cooled from  $400^{\circ}$ C to  $50^{\circ}$ C with a rate of  $-10^{\circ}$ C/min.

The complete method is displayed in figure 2.7a. For each treated laminate two specimens were tested, one from the top part of the laminate and one from the bulk of the laminate, a schematic representation is displayed in figure 2.7b. The samples were chosen this way because the top of the laminate was exposed to oxygen, which is an accelerator for degradation. A reference sample has been measured as well, in order to be able to compare the results. The second test set is not analysed with the DSC.

In figure 2.8 a cooling trace of a DSC measurement is shown. The specific area of interest is the peak around  $290^{\circ}$ C, the area under the peak is a measure for the crystallinity content. Which is together with the peak temperature an indicator for degradation.

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Figure 2.7: DSC measurement overview.



Figure 2.8: DSC cooling trace.

#### 2.5 3-point bending

3-point bending tests were performed according to ASTM D790-03 in order to evaluate the mechanical performance of the stamp formed laminates. Five specimens were cut out of each laminate and sized according to the measurements in figure 2.9b (first test set) and 2.9c (second test set). The specimens were placed on the supports as displayed in figure 2.9a, this ensures a polymer dominated fracture behaviour. Furthermore the to oxygen exposed top of the laminate was placed down to be able to measure the possible degradation of the laminate. Before performing the 3-point bending test, the exact sizes of each specimen were measured and the maximum stress at the outer layer ( $\sigma$ ) was determined with equation 2.

$$\sigma = \frac{My}{I}; \quad M = \frac{Pl}{4}; \quad I = \frac{lh^3}{12} \tag{1}$$

With y to be equal to  $\frac{1}{2}h$ , combining these equations yield:

$$\sigma = \frac{P \cdot l \cdot \frac{1}{2} \cdot h \cdot 12}{4 \cdot l \cdot h^3} = \frac{3P}{2h^2}$$
(2)

With P equal to the maximum load and h equal to the laminate thickness. P was determined depending on whether the specimen failed or not:

- For a failed specimen *P* was determined as the maximum load for the first crack.
- Otherwise *P* was determined as the maximum load of the measurement within a displacement of 0 to 7 mm.

#### First test set

The first test setup had a test speed of 1 mm/min and a support span of 37.09 mm. Of each treated laminate five specimens were cut and tested as described.

#### Second test set

For the second test seat the test speed was increased to 2 mm/min and the support span was measured at 36.80 mm. Of each treated laminate five specimens were cut and tested as described. Separately a few specimens from the first test set were tested for the influence of the test speed.







This section contains the results and short discussions of these results. First the thermocouple measurements during the stamp forming process are presented. This is followed by the results of the differential scanning calorimetry experiments and ends with the results of the 3-point bending experiments.

#### 3.1 Stamp forming (heat treatment)

During the stamp forming process the temperature development within the laminate was monitored with thermocouples. The next part contains the thermocouple results of the first and second test set.

#### First test set

For the first test set the laminates were embedded with two thermocouples: one in the middle of the laminate and one under the top layer. Figure 3.1 displays the thermocouple data of one laminate. From this figure it is clear that the temperature difference within the laminate is negligible. Figure



Figure 3.1: TC comparison

3.2 displays the thermocouple measurements of three laminates, for each test condition one. The thermocouple measurements show a clear distinction between each test condition:

- 1.  $445^{\circ}$ C for 95 seconds.
- 2.  $480^{\circ}$ C for 55 seconds.
- 3.  $480^{\circ}$ C for 150 seconds.

#### Second test set

For the second test set the laminates were embedded with only one thermocouple in the middle of the laminate. Unfortunately only one of the embedded thermocouples worked during the stamp forming

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Figure 3.2: TC conditions

procedure, therefore another thermocouple was placed on top of the laminate. Figure 3.3 displays the thermocouple data of one laminate with a thermocouple in the middle of the laminate and one placed on top. From this figure it is clear that the temperature difference within the laminate converges to a difference of about 15°C, but as measured previously, see figure 3.1, it is expected that the difference within the laminate is less than 15°C. Figure 3.4 displays the thermocouple measurements of each test condition. The thermocouple measurements show a convergence to about 380/390°C.







Figure 3.4: TC conditions

#### 3.2 DSC

The DSC apparatus measures the heat flow needed in order to increase or decrease the temperature of a sample. From this measurement a curve can be plotted, such a curve is plotted in figure 3.5. The bottom part of this curve is the heating trace and the top part the cooling trace. From the



Figure 3.5: DSC curve of the reference sample

peak in the cooling part of the curve relevant information about the crystallinity content and the peak crystallisation temperature can be determined. Figure 3.6 shows the relevant peak of the reference sample and of a top and bulk sample. From this plot it is easy to see a change in shape and position of the peak, the change of shape is an indication of a change in crystallinity and the position a change in peak crystallisation temperature. For all samples the crystallinity content and peak crystallisation temperature were determined and averaged for each heat treatment condition. These results are presented in figure 3.7, both the average crystallinity, figure 3.7a, and the peak crystallisation temperature, figure 3.7b, show little to no change for all heat treatment conditions in comparison to the reference sample.



Figure 3.6: DSC curve of the reference sample and of a top and bulk sample.



(b) Average peak crystallisation temperature.

**Figure 3.7:** Average DSC results, obtained from 1 reference sample, 3 laminates per test condition and of each laminate 1 top sample and 1 bulk sample have been measured.

#### 3.3 3-point bending

The results are averaged for each test condition and will be discussed for each test set. The averaged results however are only the result of non failed specimens, the failed specimens are presented and discussed separately because there is no clear tendency across the test sets.

#### First test set

The 3-point bending setup measures the force at a certain displacement. This force and displacement is transformed to the maximum stress in the outer layer and the flexural strain to have a measure which is independent of the specimen size. In figure 3.8 the stress/strain curves for laminate 10 ( $480^{\circ}$ C for 150 s) are displayed. All specimens passed the 3-point bending procedure except for one, specimen 1 failed at a flexural strain of 14 with a stress of 40 MPa, the failure behaviour was characterized by transverse cracks in the outside bend. Similar observations were made for all specimen sets: per condition 3 laminates are tested and each laminate was divided into 5 specimens, leading to a total of 15 tested specimens per condition. The amount of specimens per test condition that failed are:

Condition 1: 1 specimen failed.

Condition 2: 0 specimens failed.

Condition 3: 2 specimens failed.

showing there is no clear tendency across the specimen sets with respect to laminate failure. The



Figure 3.8: Stress - strain curves of laminate 10 (480°C for 150 s).

maximum (normal) stress in the outer layer was determined for all specimens and are averaged for each heat treatment condition. The average results of the first test set are displayed in figure 3.9,

these results only take the non failed specimens into account. The results of the reference measurement are the maximum average stress at first crack, but because these samples were already bend and contained transverse cracks on the outside bend comparison of other measurements to the reference material is not possible. More about this matter is discussed in the discussion, section 4.3.



**Figure 3.9:** Average results of the maximum stress of the first test set, for each condition 3 laminates divided in 5 specimens are tested, failed specimens are not taken into account for the average maximum stress. Condition 1: average of 14 specimens, condition 2: average of 15 specimens and condition 3: average of 13 specimens. Reference measurement is the average maximum stress at first crack, average of 5 specimens.

A second measurement with some specimens of the first test set was performed at which the test speed was increased fro 1 mm/min to 2 mm/min. This was done because Kumar [4] tested at 2 mm/min as well. The measurement showed a slight increase in maximum stress of about 10 MPa.

#### Second test set

The second test set was analysed the same way as the first test set and the results were again averaged for non failed specimens, see figure 3.10. Per condition 1 laminate is tested and each laminate was divided into 5 specimens again. The amount of specimens per test condition that failed are:

Condition 1: 0 specimens failed.

Condition 2: 2 specimens failed.

Condition 3: 0 specimens failed.

again showing no clear tendency across the specimen sets with respect to laminate failure.



**Figure 3.10:** Average results of the maximum stress of the second test set, for each condition 1 laminate divided in 5 specimens are tested, failed specimens are not taken into account for the average maximum stress. Condition 1: average of 5 specimens, condition 2: average of 3 specimens and condition 3: average of 5 specimens.

### 4 Discussion

The results of the research are discussed beginning with the DSC results followed by the 3-point bending results. This is followed by a discussion of other findings which are observed during the research.

#### 4.1 DSC

The DSC measurements show a slight change in the area of the cooling crystallisation curves accompanied by a slight shift of the peaks. However further analysis of the cooling crystallisation curves show that the area of the curves are almost unchanged, therefore the crystallinity of the treated laminates is more or less the same with a crystallinity content of about 34%. In comparison to previous research conducted by Kumar [4] these results are not unexpected. Kumar found for a hold time of 10 minutes a decrease in crystallinity to a content of 22% (for  $445^{\circ}$ C) to about 30% (for  $385^{\circ}$ C).

The through thickness analysis shows some discrepancies because some bulk samples of the treated laminates show a slight increase of crystallinity content. These results can possibly be due to the analysis method. The method depends on how and where the baseline for the calculation of the area under the crystallisation peak is placed. Slight variations in placement cause a difference in calculated crystallinity content.

#### 4.2 3-point bending

During the 3-point bending measurements only several specimens failed, showing no clear tendency in relation with the heat treatments and do not indicate degradation. There is no clear tendency in the average maximum stress for the first test set as well. Although these measurements are done with a crosshead displacement rate of 1 mm/s, several samples of this set were tested again with a rate of 2 mm/s to be able to compare the results to previous research. The test with the higher crosshead displacement show an increase of the average maximum stress of about 10 MPa. Comparing to the research performed by Kumar [4] it is expected that for the heat treatments of 445°C for 95 seconds and 480°C for 55 seconds the laminates do not fail, however a lower average maximum stress is observed, which is unexpected.

Reference measurements can not be used for comparison because the samples were already bend. Furthermore the samples of the reference material showed transverse cracks on the outside bend, leading to a measurement of poor quality specimens. More about the bend and transverse cracks in the reference material is discussed in section 4.3

For a second set of measurements laminates are treated with an oven temperature of  $445^{\circ}$ C for 95, 240 and 480 seconds. The 3-point bending measurements are performed with a crosshead displacement rate of 2 mm/s and the average maximum stress for all three heat treatments is about 80 MPa. This is slightly lower than Kumar [4] observed for a treated laminate of  $385^{\circ}$ C for 10 minutes, while the laminate temperatures of this test set were more or less the same.

#### 4.3 Other findings

Other findings during the research are related to the base laminate used for this research. The discussion is split into the laminate itself and the layup of the laminate.

#### Laminate

After the production of the base laminate, the laminate was cut into pieces. But during the cutting of the first laminate, the laminate bended, see figure 4.1. The laminates produced in the picture frame showed the same bending behaviour when the edges were trimmed off. The bend that arose caused transverse cracks, see figure 4.2, on one side, leaving it unsuitable for reference measurements in the 3-point bending procedure.



Figure 4.1: Bended base laminate.

This bending was probably caused by a temperature difference during the cooling sequence between the upper and lower part of the press. Appendix A shows the temperature measurement of both the upper and lower press plate (blue) and the temperature difference (red) of the three production cycles. During all three cooling periods the upper press plate tended to be cooler than the lower plate, probably causing the upper parts of the laminates to shrink faster.

Possibly the main reason for the laminates to bend this easily is the layup of the material. Although it is symmetrical ( $[0_3/90]_s$ ), the laminate is not balanced causing a lack of stiffness in the  $90^\circ$  direction.

Because the laminate consists of only 8 plies the laminate was only 1.1 mm thick and heated almost uniformly in quite a short time. A thicker laminate may cause a larger temperature distribution

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Figure 4.2: Transverse cracks on outside bend.

over the laminate thickness and will need the laminate to be heated for a longer time until the whole laminate reaches the processing temperature.

#### Layup

The layup used in this research is not only a cause for concern with respect to the bending stiffness of the reference material, but for standard mechanical testing as well. A  $[0_3/90]_s$  layup is not standard and as mentioned previously very prone to bend. But another downside to this layup is that it is not possible to compare (mechanical) measurement data to standard material data.

### **5 Conclusion and recommendations**

In this research the possible degradation of 8-ply UD C/PEEK during the stamp forming process is investigated. This was done by means of different IR oven temperatures and heating times. This section summarizes the major findings and conclusions which are followed by some recommendations for further research.

The degradation of the polymer is quantified by measuring the crystallinity content. Measurements of the crystallinity showed no significant reduction for the treated laminates which indicates that 8-ply UD C/PEEK laminates do not degradate with the chosen heating settings during the stamp forming process. Furthermore, some laminate samples showed a higher crystallinity in the bulk of the material in comparison to its surface and the untreated material. It is possible that this is due to the place in the treated laminate where the sample is obtained and a possible change in cooling rate caused an increase in crystallinity.

3-point bending tests are performed to measure the performance of the polymer matrix after stamp forming. The 3-point bending tests show no significant decrease in polymer matrix performance.

This research showed that the heating settings used for the 8-ply UD C/PEEK laminates to reach a processing temperature of  $385^{\circ}$ C no degradation occurs. However, the usability of this research is limited and showed some flaws in the process, therefore some recommendations for further research are proposed:

- With only 8 plies the laminates are relative thin and fairly easy to be heated. Thicker laminates
  will need longer heating times causing the outer layers to be exposed to heat for a longer time. A
  study to the heating behaviour of thicker laminates and the crystallinity of the thicker laminates
  will gain more insight in degradation during the stamp forming process.
- The layup used for this research is not standard and proved to be prone to bending. It is recommended to use a more standardized layup, such as  $[(0/90)_x]_s$ , for further research. The use of a standardized layup ensures a better reference material for mechanical tests and gives the possibility to compare the material performance to material supplier datasheets.

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### **A Temperature cycles**

The following figures show the temperatures of the upper and lower press plates during the production of the laminates. The temperature difference between the upper and lower press plates is calculated and plotted in the same figure. For the  $600 \times 600$  laminate the average of all 9 heating zones was taken. For the pictureframe laminates only zone 5, the middle of both press plates, was taken into account because this zone has the most effect on the heating and cooling of the laminate during the consolidation cycle.



Figure A.1:  $600 \times 600$  laminate press temperatures, blue, and temperature difference, red.



Figure A.2: Pictureframe laminate press temperatures zone 5, blue, and temperature difference, red.



Figure A.3: Pictureframe laminate press temperatures zone 5, blue, and temperature difference, red.

## **B** Reflection on the internship

#### B.1 On the employer

The Thermoplastic composites Research Center (TPRC) is an organization which is dedicated to the research and development of thermoplastic composites and its production processes for the industry. The aim of the research is to increase the use of thermoplastic composites as a lightweight material in the industry. This is because the research is funded by partners from the industry as well as academic institutions. This causes a diversity in research and mostly a direct link to challenges observed by the industry. The great diversity also ensures different people to have different expertises which in turn ensures that there is always someone who can help you with your challenges during your research.

#### **B.2** Reflection

At first the assignment was quite broad and had to be narrowed down to an achievable assignment within 4 months, the result was a continuation on previous research which was more specified to a particular production process. This causes less freedom in choices from the material to the experimental methods used. Unfortunately the results were less exciting than I hoped when I started the assignment.

The internship had a great variety from my own assignment up to all sorts of other small tasks. These small tasks suited me quite well and ensured that I was not only working on my assignment. I also liked the variety within my assignment where I had to figure out some theoretical difficulties but also had to make my own test samples and test them. In the future I would like to see this kind of variety in my career.

During the final phase of my internship it was difficult to estimate the amount of work that had to be done and how much time was needed to complete it. This made it difficult for me to make set deadlines and keep myself to them.

Eventually my internship have given me the insight of what I want to do after my study. It has become clear that my choice for the Design and Construction profile was a step in the right direction of becoming an engineer and not an all in out researcher.