COMPRESSIBILITY AND RELAXATION CHARACTERISTICS OF BINDERED NON-CRIMP-FABRICS UNDER TEMPERATURE INFLUENCE

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ABSTRACT

This study examines the compression and long-term relaxation behaviour of bindered textiles at elevated temperature levels. Experiments were conducted in a specifically designed compressibility test-rig on a carbon fibre non-crimp fabric with Epoxy resin binder. It was found in series of loading-relaxation-unloading tests, that the test temperature level significantly influences the maximum compaction pressure during the loading stage as well as the pressure characteristics during the relaxation stage. Furthermore, it was found that a significant change in the compression behaviour occurs well below the specified processing temperature of the binder. The findings of this work are intended to support optimizing preforming and preform handling steps for liquid composite moulding processes.

1. INTRODUCTION

1.1 State-of-the-art

In recent years, fibre reinforced polymer composite materials are used in an increasing number of applications and industrial sectors [1]. With the growing expertise in handling and processing this type of material, the geometric complexity of the parts was raised as well. In particular, resin transfer moulding (RTM) allows for producing parts meeting high requirements on mechanical properties, surface quality as well as geometric tolerances. However, highly stress resistant parts require a high level of fibre volume fraction (FVF). Increasing the FVF can lead to complexities in the preforming process as was well as to difficulties while placing the preform in the RTM mould [2], which in turn is crucial for the subsequent impregnation of the preform and the overall part quality.

1.2 Motivation

Based on the necessity to reduce production waste and increase process capabilities, this study addresses the compaction and relaxation behaviour of bindered textiles at elevated temperature levels. This will help to raise fundamental knowledge about the material behaviour and thus, to improve preforming performance for these types of materials. The work at hand is intended to close the existing gap of knowledge between the short term compaction behaviour, extensively reported in scientific literature [3,4], and the more process-relevant long-term compaction behaviour.

2. EXPERIMENTATION

2.1 Materials

For this study on compaction and relaxation behaviour of bindered textiles, a biaxial carbon fibre non-crimp fabric (CF-NCF) with 555 g/m² fibre area weight (FAW) was used. Included in the FAW is a 6 g/m² polyethersulfone (PES) stitching yarn as well as an Epicote 05311 epoxy resin binder, applied on one side of the textile to 5-wt.% as a powder, with a specified processing temperature of 102 °C. Each specimen consisted of seven 120 x 120 mm² layers, which were cut by a rotating blade on a ZÜND automatic cutting system, ensuring clean cuts and avoiding fibre pull-out. The layers were uniformly stacked, with the binder on the upper side, resulting in a stacking order of $[-45/\underline{+45}]_7$ and weighed just before being placed on the test area, to reduce unnecessary handling and influence in the test result. In total, 63 specimens were prepared in six groups, with very little overall inhomogeneities (e.g. missing rovings, inhomogeneous binder application, etc.) in the textile, as shown in Table 1.

Table 1: Overview of the average FAW of the specimen and their respective coefficient of variation (c_v: standard deviation divided by arithmetic average). As indicated, the samples show only little inhomogeneities, weight and structure wise.

Test group	Average FAW [g/m²]	c_v (FAW) [%]	Number of Specimen [-]
V1.X	554.83	0.17	11
V2.X	555.18	0.27	10
V4.X	554.54	0.27	11
V6.X	554.55	0.22	11
V7.X	555.38	0.20	10
V8.X	555.31	0.16	10
Total	554.95	0.23	63

2.2 Novel Test-rig for compaction measurements

Based on the findings of previous research [5,6] a new test-rig was designed to fit the needs of dry as well as wet compaction tests at elevated temperature levels. For this study, the set-up for dry compaction tests was used, as detailed in Figure 1.

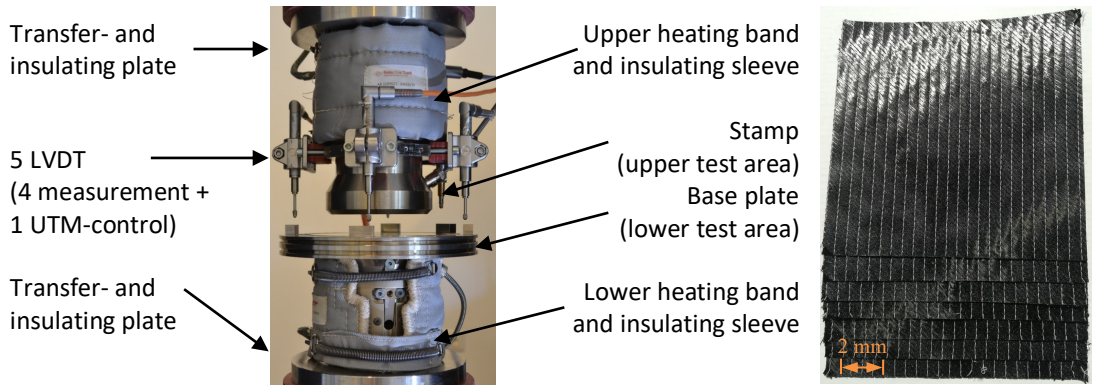

Figure 1: Compression test-rig (left) and specimen tested in loading relaxation unloading tests (right).

The stamp diameter of 100 mm allows for large specimen to be tested, reducing the effects of local inhomogeneities in the material. The active heating through heating bands placed at some distance from the upper and lower test area allows for test temperatures of up to 250 °C, while at the same time not influencing the thermally decoupled linear variable displacement transformer (LVDT) used for machine control and thickness measurement of the specimen. The heating process is controlled by a specifically developed heat pulsing software, supported by two thermocouples located closely to the upper and lower test area, respectively. The software ensures temperature consistency of the test area of less than +/- 1 K from the temperature set point.

2.3 Test method

Based on previous research on the topic of compaction measurements a common test scheme was used for all presented results. This scheme, as detailed in Table 2, follows the example of a "loading-relaxation-unloading" test configuration as it was for instance used by [3,4] and also in the latest benchmark on compaction measurements [5]. A pre-heating stage was added to the scheme in order to ensure homogeneous specimen temperature, which was monitored by a thermocouple placed between the third and fourth layer of the stack. After establishing loose contact (compaction pressure of less than 2000 Pa), the crosshead position was frozen for five minutes to heat the specimen up to the desired test area temperature. Subsequently, the specimen was compressed to a target stack height of 3.721 mm, which corresponds to a nominal level of FVF of 0.58.

Table 2: Process steps and control settings of the relaxation measurement

Process step	Crosshead position [mm]	Crosshead speed [mm/s]	Duration [s]
Closing	–	50	–
Establishing contact	–	5	–
Pre-heating	5.45	–	300
Loading phase	–	1	–
Holding phase	3.721	–	3600
Unloading phase	–	-1	–
Opening	–	-50	–

After reaching the target stack height, the crosshead position was held constant for one hour for the relaxation stage of the test, before unloading the specimen.

3. RESULTS

Data obtained in previous studies, e.g. [7–9], showed a strong temperature influence on pressure development in compaction measurements with bindered textiles. While most studies were focussing on the loading part of the compaction process or only a few minutes of the relaxation period, the study at hand addresses the compaction and relaxation behaviour of a bindered CF-NCF over a much longer period and at different temperature levels.

3.1 Compaction behaviour of bindered textiles at different temperature levels

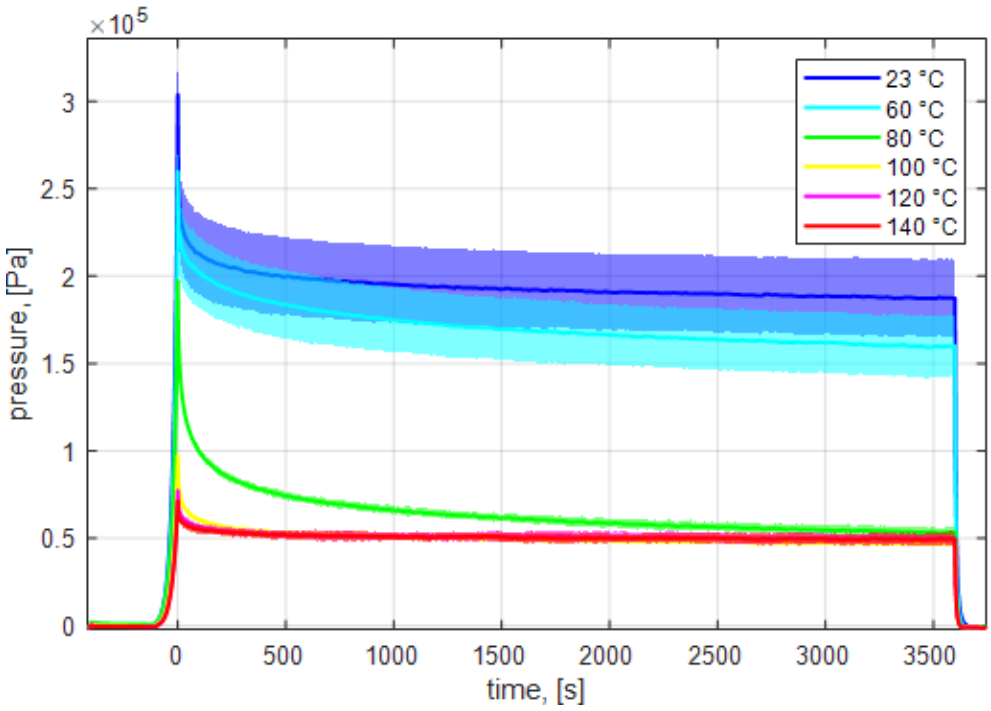

Figure 2: Comparison of mean compaction pressure with standard deviation envelopes of all temperature levels for one-hour relaxation test procedure.

As Figure 2 shows, after a rapid increase of compaction pressure and reaching a pressure maximum (p_{max}), the compaction pressure rapidly drops, due to reorientation and nesting effects in the specimen, as e.g. previously shown in [4] and [10]. For all temperature levels the initial rapid pressure drop slows down significantly after about 200 s. After about 500 s (for temperature levels of 100 °C and above) and 1500 s (for temperature levels of 80 °C and below), this behaviour changes to an almost linear behaviour, before reaching steady state conditions. Post-processing the results, p_{max} and p_{end} values were identified to compare the relaxation behaviour at the different temperature levels. p_{max} refers to the maximum pressure during the compaction test, while p_{end} was defined as the average pressure over the last 60 s of the relaxation period.

A more detailed examination of the results showed that the steady state conditions could not be reached for temperature levels of 80 °C and below. This behaviour is most prominent for the tests at 60 °C, where a decrease of compaction pressure is still visible on the one-hour time-scale. Due to this unexpected result, additional tests with extended, long-term relaxation periods were conducted.

Table 3: Overview of maximum pressure (p_{max}) and end pressure (p_{end}) for all temperature levels together with their respective coefficient of variation (c_v).

Test group	Temperature level	Average (p_{max})	c_v (p_{max})	Average (p_{end})	c_v (p_{end})	Number of tests
	[°C]	[10^5 Pa]	[%]	[10^5 Pa]	[%]	[-]
V1.X	23	3.04	9.99	1.88	11.19	11
V2.X	60	2.65	10.54	1.61	9.42	10
V4.X	80	1.98	2.88	0.54	5.38	11
V6.X	100	0.97	3.80	0.47	3.61	11
V7.X	120	0.77	3.41	0.49	4.50	10
V8.X	140	0.71	3.71	0.50	7.54	10

Table 3 shows the results of the one-hour loading-relaxation-unloading tests. Above a temperature level of 80 °C, the maximum compaction pressure drops significantly, as does the coefficient of variation. In order to study the amount and speed of compaction pressure drop, normalized compaction pressure characteristics (i.e. compaction pressure normalized by its maximum) are visualized in Figure 3.

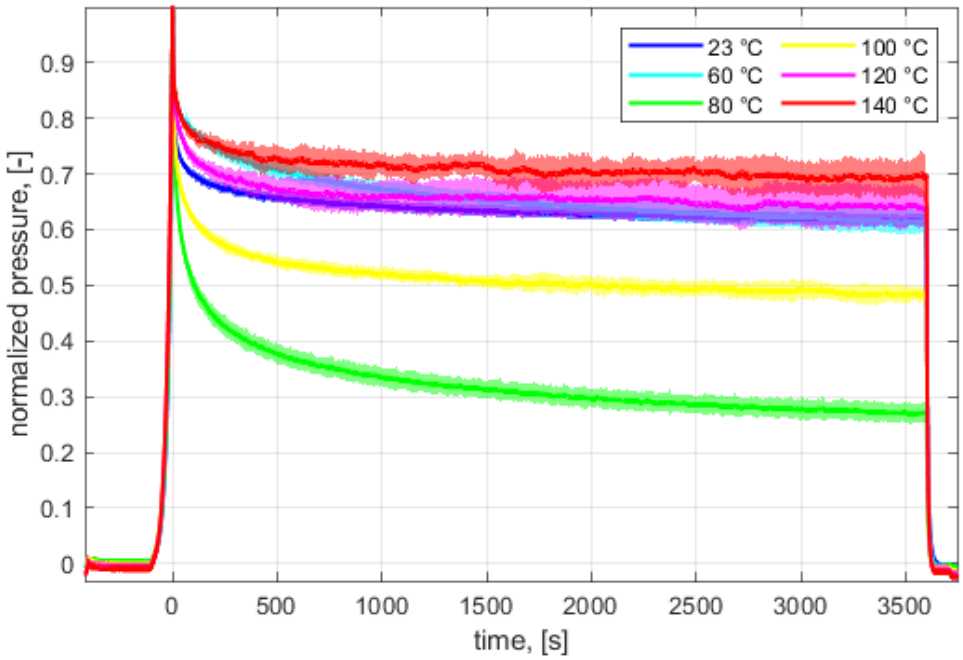

Figure 3: Comparison of normalized pressure (average characteristics together with standard deviation envelopes) of all temperature levels for one-hour relaxation test procedure.

The normalized characteristics show similar trends for tests conducted at 23 °C, 60 °C, 120 °C and 140 °C, while tests at 80 °C and 100 °C show strongly deviating trends. The highest amount of relative pressure drop was found at 80°C, which is well below the proposed processing temperature specified in the technical material data sheet.
Table 4 gives a quantitative overview of absolute and relative pressure drop, respectively, together with the corresponding coefficients of variation.

Table 4: Absolute and relative changes in compaction pressure of the our-hour relaxation measurements.

Test group	Temperature level	Average pressure drop	c_v (pressure drop)	Relative pressure drop	Number of tests
	[°C]	[10^5 Pa]	[%]	[%]	[-]
V1.X	23	1,16	8,33	38,32	11
V2.X	60	0,99	14,97	39,03	10
V4.X	80	1,43	4,87	72,76	11
V6.X	100	0,50	7,05	51,13	11
V7.X	120	0,28	10,72	36,06	10
V8.X	140	0,22	10,55	30,26	10

As can be seen in this comparison the highest relative changes due to relaxation occurred at temperatures of 80 °C and 100 °C. The least distinct relaxation behaviour was found at 140 °C, while being in similar range with the remaining results at 23 °C, 60 °C and 120 °C respectively.

In addition, Figure 3 indicates that after one hour of relaxation time, steady state pressure characteristics were only found for tests at 120 °C and 140 °C. In particular, the tests conducted at 60 °C and 80 °C show continuous pressure drop for the entire duration of the relaxation period. In order to analyse this effect, additional long-term tests were conducted.

3.2 Long-term compaction behaviour

Figure 4 shows the results of two long-term tests with a relaxation period of 12 h for the test at 60 °C and 8 h for the test at 80 °C, respectively.

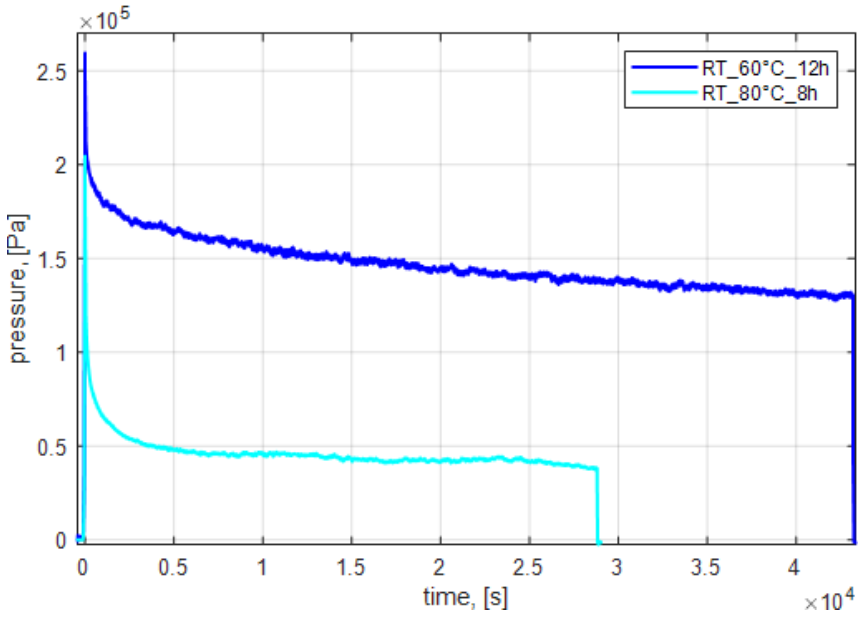

Figure 4: Comparison of long-term relaxation behaviour at temperature levels of 60 and 80 °C.

As can be seen, after 8 and 12 hours of relaxation time, respectively, no true pressure equilibrium could be reached. Absolute changes in compaction pressure were low for both

tests, but added up to a non-negligible amount over the long test time. Compared to the tests at 60 °C, the linear decrease of compaction pressure at 80 °C is significantly slower. This could be seen as an indication, that the time necessary to reach an equilibrated level is shorter for 80 °C than for 60 °C.

Measurement errors or drifts in the load cell signal can be ruled out, since blind tests (direct contact of the compression stamp with the base plate) up to 2 kN and 15 kN were carried out at the beginning and end of each test day, showing a good linearity with less than 0,0015 mm internal test-rig deformation at 15 kN. In addition, an error due to temperature variation can be ruled out, since a temperature equilibrium inside the specimen can be measured after less than five minutes of test time, and the ambient temperature can be regarded as constant during the tests.

4. CONCLUSIONSAND FUTURE WORK

Prior work has documented the relaxation behaviour of textiles as well as the changes in compaction behaviour due to an influence of temperature on bindered textiles. However, these studies have focused only on the short-term behaviour, e.g. the initial seconds or minutes after start of the compaction. In contrast to this, this study looks at the wider picture and expands on previous findings through analysis of the relaxation behaviour at different temperature levels during the first hour of compaction and beyond. It was found that besides increasingly lower maximum compaction pressure levels for rising temperatures, confirming prior work on short term compaction behaviour, the most significant change occurs at a temperature level well below the proposed processing temperature of the binder. Furthermore, an unusual long-term relaxation behaviour (no constant pressure level) was documented for temperature levels between room temperature and binder activation temperature.

4.1 Relaxation behaviour below binder activation temperature

As Figure 2 showed, a general decline in p_{max} can be observed for increasing temperature levels. In addition, it was found that a significant drop in p_{max} occurs between temperature levels of 60 and 80 °C as well as a change in pressure progression during the relaxation stage. Figure 3 showed, that the lowest pressure drop ratio (p_{end}/p_{max}) can be observed for 80 °C, 20 K below the proposed processing temperature of the binder. Based on this finding, further measurements will be conducted to locate the temperature level at which a significant decline on compaction pressure initially occurs and at which the pressure drop ratio reaches a minimum. Moreover, additional research has to be conducted on the topic of long-term relaxation behaviour below binder activation temperature. This includes additional measurements at different temperature levels below binder activation temperature, but also longer test periods. The aim of the extended long-term tests will be to (i) find the time to reach equilibrium pressure at temperature levels below binder activation temperature and (ii) to find the remaining pressure level once an equilibrated state is reached.

4.2 DSC and viscosity measurements

Additionally, differential scanning calorimetry (DSC) tests will be performed on Epoxy binder material. As provisional results have shown, changes in relaxation behaviour appear well below the binder activation temperature specified in the technical material data sheet. The DSC

measurements will be used to examine the glass transition temperature (T_g) of the binder system and the long term-behaviour at elevated temperatures. This will clarify, whether unaccounted effects underly the results of the long-term measurements that could explain the illustrated results. Furthermore, viscosity measurements will examine the temperature level at which the mechanical properties of the binder system change in such a way, that contributes to a "lubrication effect". This in turn will also help to link the measured behaviour to the binder itself or to an interaction between the binder and the textile. In either case, it will lead to a better understanding of material behaviour and help to optimise preforming parameters, like compaction pressure and preforming temperature. Implementing those ideal preforming parameters will result in improved preform processing and handling, which will boost production output and increase part quality.

5. ACKNOWLEDGEMENT

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