POWER ULTRASONIC IN CLOSED INJECTION PULTRUSION

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Abstract
The aim of this work is to characterize the influence of power ultrasonic in combination with closed injection pultrusion to increase the impregnation quality. For this purpose, two examination methods are used. On the one hand the influence of power ultrasonic on the resin system is investigated in the laboratory. The amplitude, the sonication time and the pressure are varied as manipulated variables. On the other hand the power ultrasonic is integrated into an injection and impregnation chamber upstream of a pultrusion die. This allows the power ultrasonic to be examined under process conditions. The position and the amplitude are varied as manipulated variables. The results provide important data to understand the influence of power ultrasonic to closed injection pultrusion.

1. Introduction

Pultrusion is a highly automated production process. The fiber volume content usually ranges from 65 to 70 % and is at the upper end of the technically feasible. For high-performance components, the conventionally used glass fibers are replaced by carbon fibers. Carbon fibers have a lower permeability and are relatively difficult to impregnate with resin system [1,2]. The impregnation of the fiber package during manufacture is not an issue for a large part of the profiles on the market. In this case, an open bath system such as dip or pull-through bath is used for impregnation. A complete impregnation is guaranteed. However, a high emission of resin vapor is released into the environment. In addition, a long processing time of the resin system that lasts several hours is necessary. New innovative resin systems with highly reactive compounds, however, have a much shorter processing time lasting only a few minutes. They must be processed through a closed injection and impregnation chamber (II-chamber) with direct resin injection [3]. Even hollow profiles, consisting of braided tubes, require an II-chamber for impregnation. Nevertheless, thick-walled components impregnated by II-chamber have a high pore content.

By means of targeted coupling of power ultrasonic into the pultrsion process, the impregnation should be improved. The impregnation describes the wetting and impregnation of the fiber package with the matrix system. Until now the impregnation of the fiber package takes place by using an open bath system or different geometries of impregnation chambers. Each system has its own disadvantages and challenges [4,5]. By coupling in power ultrasonic, the impregnation of the fiber package should be facilitated. By means of the high-frequency oscillation, the fibers and the matrix are moving fast. This should ensure the complete impregnation of even strongly anisotropic fiber packages of carbon fibers with the matrix system.

2. Materials and methods

The influence of power ultrasonic on the pultrusion process is investigated by two methods. For one thing the resin characterization is implemented by a laboratory setup. The aim is to determine to what extent the resin system is changed by the power ultrasonic. At the same time the process characterization is done by a modified pultrusion process. The power ultrasonic is integrated directly into the pultrusion process.

2.1. Materials

For the resin characterization, the epoxy resin system Biresin®CR141 (Sika, Germany) is used with the following composition:

Table 1. Resin system

Category	Name	Company	Parts
Resin	Biresin®CR141	Sika	100
Hardener	Biresin®CH141	Sika	90
Accelerator	Biresin®CA141	Sika	2
Internal mold release	Chemlease® IC25	ChemTrend	6

In addition to the resin system mentioned above, the direct rovings SE 3030 with 4800 tex (3B, Belgium) are used to investigate the pultrusion process.

2.2. Die design and methods

For the characterization of the resin system, a pressure chamber was designed and manufactured. It simulates the conditions in the pultrusion process as close to the process as possible. For this purpose the pressure chamber was designed for pressures of up to 6 bar overpressure. In order to visually observe the influence of power ultrasonic, the pressure chamber was made of a pressure-resistant glass cylinder. Beyond the pressure, the temperature is also measured by the resin system. Figure 1 shows the overall setup: (1) ultrasonic processor, (2) datalogger for temperature, (3) ultrasonic transducer, (4) compressed air connection, (5) pressure gauge.

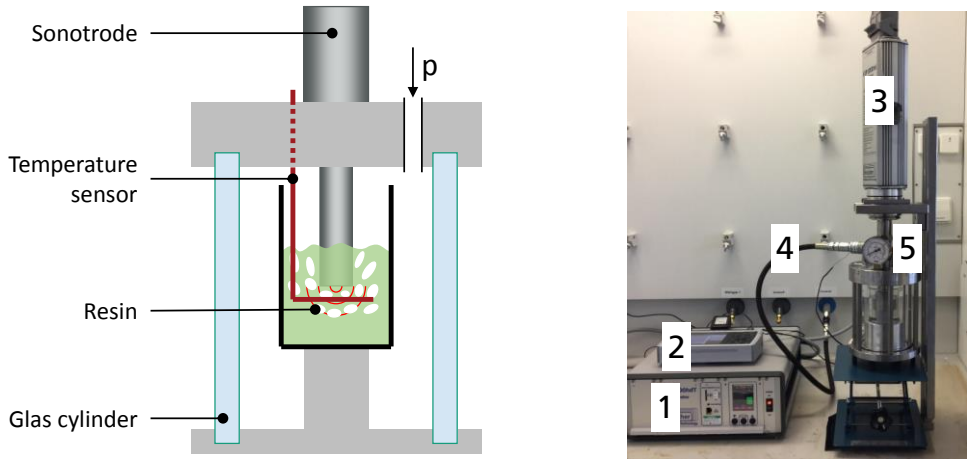


Figure 1. Pressure chamber (left) and overall construction (right)

To investigate the pultrusion process, an II-chamber was developed and used in combination with a pultrusion die (60x5 mm²). The II-chamber has 6 variable slots. 3 different inserts can be installed at 6 positions. Figure 2 shows the three possible inserts: resin inlet (TI3 and BI3), power ultrasonic (BI2), pressure sensor (TI1, TI2, BI1).

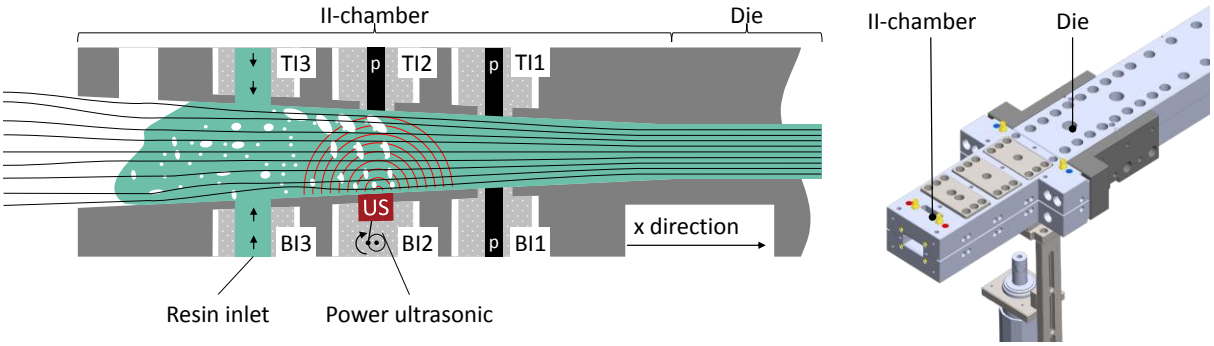


Figure 2. II-chamber (left) and overall construction (right)

2.2.1 Resin characterization

The aim of the resin characterization is to determine to what extent the resin system is changed by the power ultrasonic. The manipulated variables are as follows: pressure (0, 6 bar), amplitude (20, 40 µm) and time (10, 60 s). The examination variables are as follows: glass transition temperature T_g, viscosity at 25 °C, Fourier-transform infrared spectroscopy (FTIR) and temperature increase.

All experiments are carried out according to a fixed scheme to minimize disturbance. In Table 2 all activities are listed chronologically.

Table 2. Chronological sequence of the experiments

Time [min]	Activity
0	Weighing the resin system
	Manual mixing with wooden spatula for 1 min
	Degassing in the desiccator
20	Weighing 5 g resin system in a small beaker
40	Sonication with ultrasonic
50	Rheometer measurement
55	FTIR measurement
60	Weighing 2 samples for the DSC and turning on DSC

An overview of the experimental parameters is shown in Table 3.

T_g is determined using DSC 214 Polyma (Netzsch, Germany). The first heating rate is modeled on the pultrusion process (150 K/min, 20 ... 170 °C), followed by tempering at 140 °C for two hours. During the last heating the T_g is determined (10 K/min, 20...200 °C). The MCR 302 (Anton Paar, Germany) is used for the rheology study. The plate-plate assembly is used in oscillating mode with a pitch of 1 mm. The viscosity is determined at 25 °C ($F = 5\,Hz$, $\gamma = 5\,\%$) over the mean of three minutes. The chemical composition of the functional groups is determined by the FTIR.

2.2.2 Process characterization

The aim of the process characterization is to determine the influence of power ultrasonic on the pultrusion process. The focus here is on the extent to which the power ultrasonic coupling improves the impregnation and homogenization of the fiber package in a closed injection pultrusion process. The amplitude (0, 6.6, 13.2, 26.3 µm) and the coupling position (BI1 - TI3) of the power ultrasonic are chosen as the manipulated variable. During the process, the temperature development, the pulling force and the chamber pressure are determined. The samples were examined for their mechanical properties (ILSS and 3 point bending test).

All experiments are carried out with the same experimental setup. The die is heated with 4 zones à 180 °C. A cooling plate is mounted between the II-chamber and the die, to ensure that the II-chamber is not heated up by the die. Reference samples are made with an open bath system. Here, the number of rovings is 118. With the II-chamber, the number of rovings could be increased to 122. An overview of the experimental parameters is shown in Table 3.

Table 3. Overview of the experimental parameters

Resin characterization			
Name	Pressure [bar]	Time [s]	Amplitude [µm]
A.1	0	10	20
A.2	0	10	40
A.3	0	60	20
A.4	0	60	40
B.1	6	10	20
B.2	6	10	40
B.3	6	60	20
B.4	6	60	40
C.1	3	35	30
C.2	3	35	30
C.3	3	25	30

Process characterization			
Name	Position []	Amplitude [µm]	Speed [m/min]
xxx_00.0_0.3	Bath	0.0	0.3
TI1_00.0_0.3	TI1	0.0	0.3
TI1_00.0_0.4	TI1	0.0	0.4
TI1_06.6_0.3	TI1	6.6	0.3
TI1_06.6_0.4	TI1	6.6	0.4
TI1_13.2_0.4	TI1	13.2	0.4
TI2_00.0_0.3	TI2	0.0	0.3
TI2_00.0_0.4	TI2	0.0	0.4
TI2_13.2_0.3	TI2	13.2	0.3
TI2_26.3_0.3	TI2	26.3	0.3
BI2_13.2_0.3	BI2	13.2	0.3

To determine the temperature evolution, a thermocouple is tied to a roving and then pulled through the II-chamber and die. Thus, during the process, the temperature development in the entire structure could be determined. The pulling force is measured by an integrated load cell in the die holder. The 3 point bending test is performed according to DIN EN ISO 14125 and the ILSS according to DIN EN 2377.

3. Results and discussion

The results are presented and discussed separately according to resin characterization and process characterization in the following section.

3.1 Resin characterization

The test results are discussed in the following order: T_g, viscosity, FTIR and temperature increase.

T_g: With increasing amplitude and longer sonication time, T_g increases (see Figure 3). Otherwise, the T_g decreases with increasing pressure. Yet, the differences in the individual T_g values are small and lie in a range of 4.5 K. The scattering is comparable to the scattering of the center test C1 to C3 (2 K). In summary, the power ultrasonic has little to no influence on the T_g.

Viscosity: In the selected test procedure, the viscosity increases due to the power ultrasonic (see Figure 4). Both, the amplitude and the time increase the viscosity at 25 °C. However, as the pressure increases, the viscosity decreases. It is striking that the viscosity hardly changes in all experiments. The exception is experiment A.4, in which the resin system has the highest viscosity and experiment B.3 with the second highest viscosity. In summary, 50 minutes after sonication, the viscosity is generally increased.

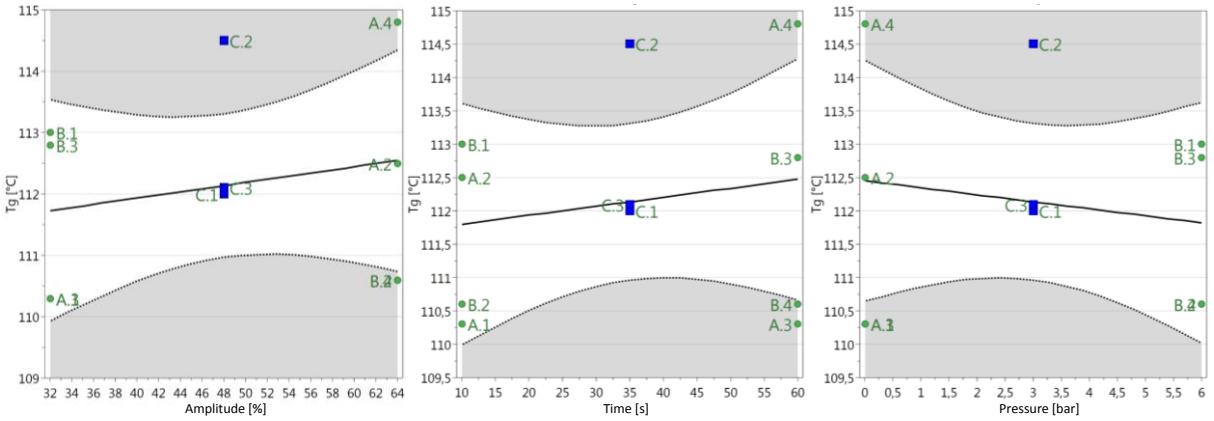


Figure 3. Effect diagram "T_g"

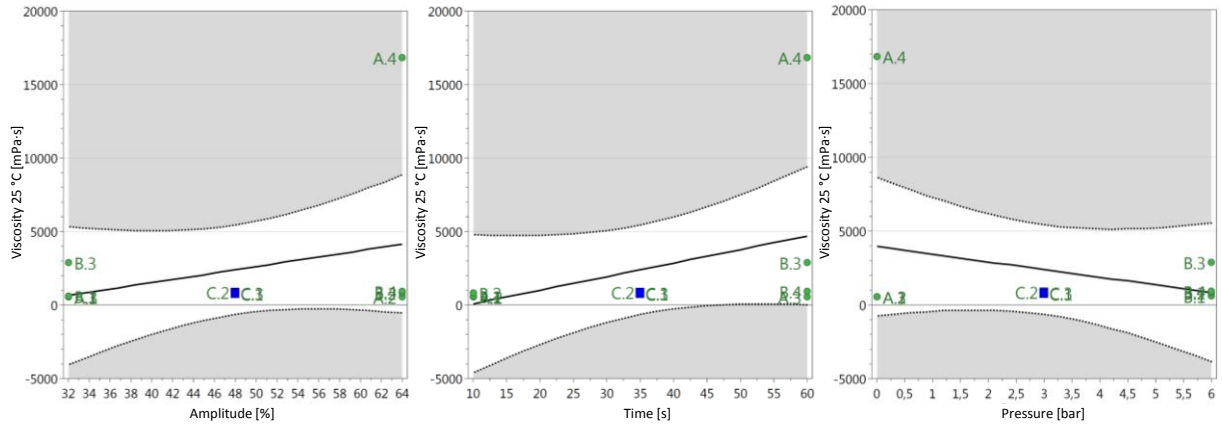


Figure 4. Effect diagramm "Viscosity"

FTIR: The reaction of the anhydride with the epoxy can be tracked in the FTIR. Thus, experiments A.4 and B.3 show the formation of the ester group which results from the reaction described. In the remaining experiments, no change in the FTIR spectrum could be detected.

Temperature increase: An increase of temperature can be observed in all experiments (see Figure 5). The temperature increase rises with higher amplitude, longer sonication time and greater pressure. To sum up the effect of the amplitude and the time in the examined settings is greater than that of the pressure.

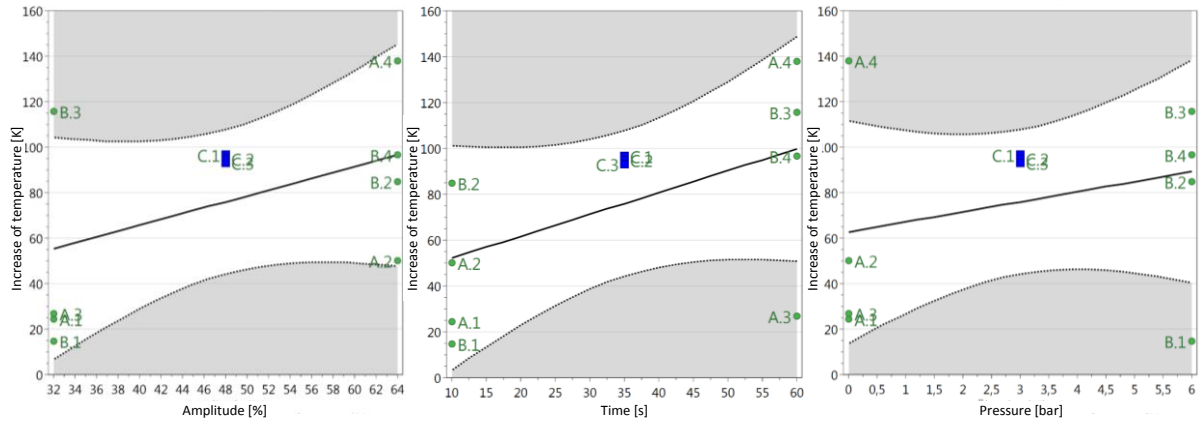


Figure 5. Effect diagram "Increase of temperature"

3.2 Process characterization

The test results are discussed in the following order: temperature development; pulling force and chamber pressure; 3 point bending test; ILSS.

Temperature development: The experiment TI1_06.6_0.4 reaches a maximum temperature of 65 °C within the II-chamber (see Figure 6). Position of the measured temperature is directly below the ultrasonic sonotrode. Experiment TI1_13.2_0.4 shows that power ultrasonic can introduce a very large amount of energy very fast into a system. Here a maximum temperature of 280 °C is measured. In experiment TI2_26.3_0.3 the maximum temperature measured is 160 °C. This is significantly lower than in experiment TI1_13.2_0.4, although the amplitude is twice as high. The hydraulic pressure increases in x-direction in the II-chamber. Thus, the pressure at position TI1 is higher than at position TI2. In summary, the results from the resin characterization are confirmed. With increasing pressure and amplitude, the highest temperature increases result.

Pulling force and chamber pressure: In TI2_00.0_0.3 the pulling force is around 9 kN (see Figure 6). The pressures are at 24 bar at position BI1 and 2 bar at BI2. The experiment represents the initial situation without power ultrasonic. It can be seen that the pressure in the x-direction increases within the II-chamber (as already mentioned in the temperature development section). With increasing compaction of the fiber package the flow resistance increases and thus the pressure build-up. The experiment TI2_13.2_0.3 shows an interesting development. After the ultrasonic was switched on, the pulling force drops to 6 kN (-30 %), the pressure at position BI1 to 7 bar (-70 %) and at position BI2 to less than 2 bar (-20 %). The trend continues when the amplitude is increased from 13.2 to 26.3 µm. A further drop in the measured values can be seen. Overall, the test series shows a reduction of the pulling force by 65 %, the pressure at position BI1 by 80 % and the pressure at position BI2 by about 100 %. All in all, power ultrasonic can achieve a considerable reduction of the pressures within the II-chamber. The reduction of pressure results directly in a reduction of the pulling force.

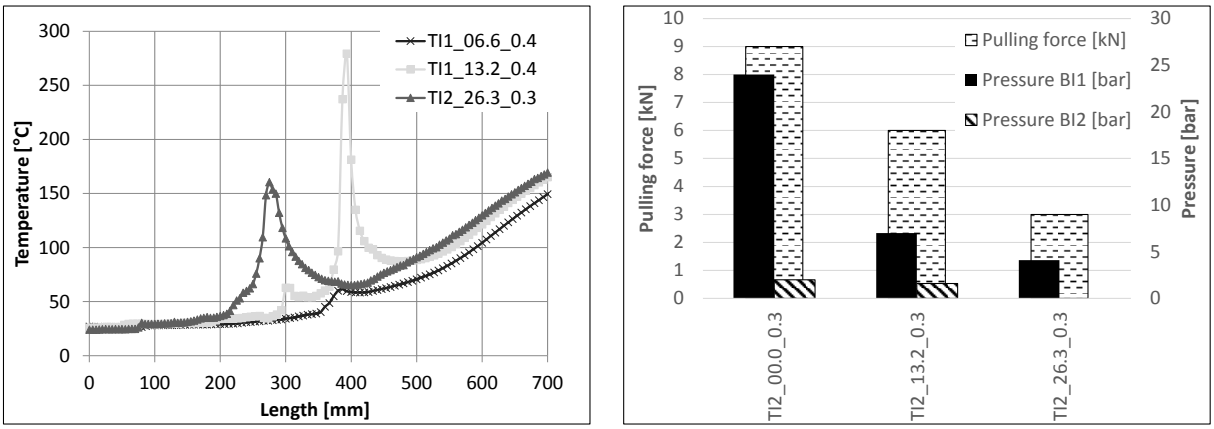
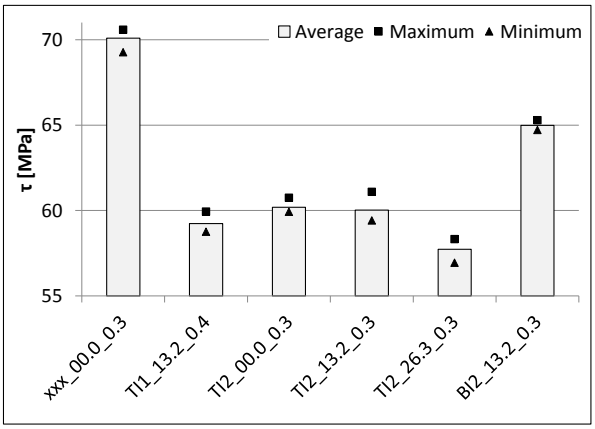


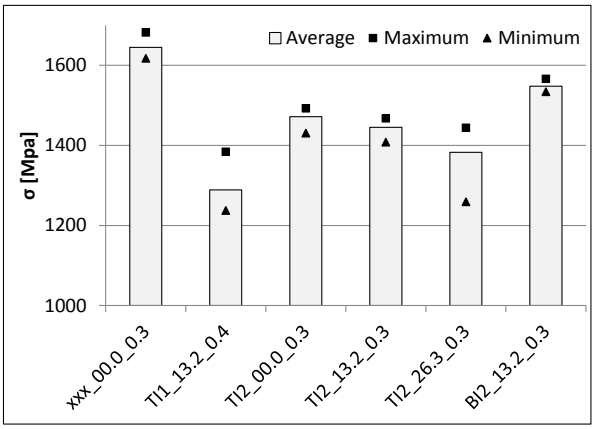
Figure 6. Temperature development (left), pulling force and chamber pressure (right)

ILSS: The experiment xxx_00.0_0.3 serves as a reference in which the impregnation is carried out by an open bath system. The shear strength is in the range of 70 MPa. All experiments with impregnation via the II-chamber do not reach the value of 70 MPa, but are in the range of 59 to 65 Mpa. Experiement BI2_13.2_0.3 has reached the highest value with 65 Mpa and also has the lowest scatter. In comparison to all experiments introducing ultrasonic from the top, it can be seen that the introduction of ultrasonic from below has a positive influence on the shear strength in comparison to a conventional II-chamber. The experiment TI1_13.2_0.4 shows a peak value of 280 °C during the temperature measurement. However, with a shear strength of 59 MPa, , the value is hardly lower than in the other II-chamber experiments. In summary, it can be said that introducing ultrasonic from below

shows the greatest positive effect. In addition, a temperature increase due to the power ultrasonic has no negative effect on the shear strength.

3 point bending test: The experiment xxx_00.0_0.3 serves as a reference. The flexural strength is in the range of 1650 MPa. All experiments with impregnation via the II-chamber have a flexural strength of 1400 to 1550 MPa and do not reach the reference value. Similar to the ILSS test, introducing ultrasonic from below also improves the properties. Compared to the conventional II-chamber (TI2_00.0_0.3), the flexural strength increases from 1475 to 1550 Mpa with reduced scattering. The experiments TI1_13.2_0.4 and TI2_26.3_0.3 have the lowest flexural strength values of 1300 and 1375 MPa with increased scattering. Especially experiment TI1_13.2_0.4 shows that high ultrasonic impact reduces flexural properties due to the fiber damage. To sum up, introducing ultrasonic from below shows the greatest positive effect. The flexural properties are better (+5%) than the conventional II-chamber. However, the flexural properties are worse (-6%) compared to the open bath system. In order to avoid fiber damage, the height of the amplitude is limited to an upper threshold.





4. Conclusion and outlook

This study examines the influence of power unltrasonic to the closed injection pultrusion process. The investigations are divided into resin characterization and process characterization. Resin characterization shows that power ultrasonic does not chemically alter the resin system, provided the ultrasonic time is not too long and intense. In addition, it can be seen that the temperature is increased by power ultrasonic. The influence on the viscosity could not be clarified in this study. The time interval between sonication and viscosity measurement was too long at 50 minutes. The process characterization shows that power ultrasonic can significantly reduce important process variables such as chamber pressure and pulling force. Power ultrasonic enables quick and intensive introduction of thermal energy into the system. On the other hand, the mechanical properties of the reference samples (open bath system) can not be achieved. Compared to the conventional II-chamber, the flexural strength can be increased by up to 5%. In further investigations, the influence on the viscosity must be examined more closely. Likewise, the investigations on carbon fibers must be extended.

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