

Research Article

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Investigation of the adhesive strength in a combined compaction and back-injection process to produce back-injected self-reinforced composites (SRCs)

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Abstract: This publication investigates the adhesion between an injection molded component and a self-reinforced composite (SRC) produced in a combined compaction and back-injection process to produce back-injected self-reinforced composites. To study the influence of the process, the parameters barrel temperature, time of injection, and tool temperature were varied. In addition, samples were taken at different positions along the flow path. In light of the orthotropic material behavior of SRCs, investigations were conducted to see whether different loading cases lead to different mechanical behavior. Shear-off and pull-off tests revealed a different strength as a function of the loading type. In the shear-off tests, a mean strength of 11.37 MPa was recorded over the entire test series, while the measured mean strength in the pull-off tests is considerably lower, 4.04 MPa. The type of failure is determined with the aid of SEM images, and the influence of the microstructure of the thermoplastic fibre materials on the adhesion is set out. It is shown that, as of a sufficiently high level of adhesion, failure occurs within the fibres.

Keywords: adhesion; back-injected; compaction; self-reinforced; SR-PP; SRC.

1 Introduction

The injection molding of thermoplastic polymers is one of the most widely employed processes in plastics manufacturing,

since it combines a high freedom of design with good cost-efficiency. Frequent use is thus made of this process in the manufacture of plastic products and, in many cases; it is combined with other processes to enable precisely these advantages to be incorporated into other processes too.

Manufacturing plastic products with continuous fibre reinforcement offers only limited freedom of design in production and is uneconomical for a large number of applications. This is due mainly to the comparatively complex process chain involved in the manufacture of these composites. It is common practice to manufacture semi-finished products as sheets, which are then formed into three-dimensional components in pressing processes. Automation of these processes has been taken forward in recent years in order to improve economic efficiency (Biermann et al. 2012, 2015; Friedrich 2017; Heim et al. 2012; Ries 2015; Rohde et al. 2014; Zimmol et al. 2012). By integrating continuous fibre-reinforced plastics in injection molding, their good mechanical properties can be combined with the freedom of design offered by injection molding. Both the final contours and functional elements can be injection molded directly onto a component in the process. This constitutes an advantage with respect to the adhesion of the injected-on components among other things, since the temperature at the interface is decisive for adhesion (Gude et al. 2017; Schuck 2009).

Both multi-component injection molding (Heim 2016; Schuck 2009) and the back-injection of organosheets (Gude et al. 2017) require a higher temperature in the interface for adhesion. With SRCs, it must be borne in mind that, due to their temperature sensitivity, the mechanical strength of the load-bearing fibres is highly dependent on their temperature exposure (Heim et al. 2013; Paßmann 2009; Ries 2015; Schöppner et al. 2013). This constitutes a challenge at the time the SRCs are produced already. Fabrics developed especially for the production of SRCs are therefore frequently made up of two polymeric phases with different melting points. The component that forms the

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matrix has a lower melting point and can thus exert a positive influence on the adhesion of an injected-on component, even with lower interface temperatures (Kmetty et al. 2010).

Scientists have already proven that the back-molding of previously consolidated SRCs is possible without the complete loss of self-reinforcement (Aurrekoetxea et al. 2006; Jerpdal et al. 2020). The investigations showed that a critical temperature has to be exceeded in the edge layer in order to achieve good adhesion. This temperature is a function of the SRC used, as is also familiar from multi-component injection molding (Aurrekoetxea et al. 2006; Heim 2016). In addition, an investigation was conducted into different temperature gradients between the SRC and the melt which lead to an identical interface temperature. This showed that larger gradients lead to better adhesion. This was attributed to a temperature peak that occurs at the interface and briefly exceeds the interface temperature that develops. It was not possible to determine any influence of the holding pressure on adhesion in the range from 247 to 371 bar. The investigations into the maximum shear stress in the interface, conducted on Curv[®] (Propex, Chattanooga, USA) and determined in tensile shear tests, showed shear stresses of between approx. 0.4 and 10 MPa for an interface temperature of between 145 and 185 °C (Aurrekoetxea et al. 2006).

Other researchers are concentrating on the temperature effect in the SRCs but have not investigated the adhesion between the injection molded plastics and SRCs. They are back-molding PET-based SRC inserts of different thicknesses with a 2 mm thick layer of melt at 265 °C, with the result that the stiffness can be reduced by up to 18% after overmolding. The resulting adhesion was not investigated (Jerpdal et al. 2020).

Investigations conducted in other previous works similarly revealed a correlation between adhesion and melt temperature. In shear-off tests on the material PURE[®] (DIT B.V., Dinxperlo, The Netherlands) that was back-molded with a PP, shear stresses between 3.26 and 5.81 MPa were measured over a melt temperature range of 160–180 °C (Jakob et al. 2021).

In this publication, the adhesion between a PP injection molded component and SRCs is investigated as a function of different parameters. The materials are joined together in a new combined compaction and back-molded process (Jakob et al. 2021). In addition to the use of different process parameters, the geometry was varied when studying the adhesion. Also, different types of loading in the form of shear-off and pull-off test were employed for the investigations.

2 Experimental studies

2.1 Materials

A special fabric developed for the production of SRCs was used for the tests. This is PURE[®] material from the DIT company which is a fabric made up of thermoplastic tapes (plain weave). The fabric is a specially modified one with film strips (tapes) that have coextruded edge layers with a low melting point. The volume ratio of core to edge layers is 5:90:5 (Ries 2015). These can be melted during hot compaction, thus forming the matrix without exposing the highly stretched core to an excessively high temperature. The fabric is 100% polypropylene and has a weight of 105 g/m². The processing temperature is specified as between 130 and 180 °C by the manufacturer. The temperature range and the effect on the mechanical properties of the SRCs has been tested and described by other scientific workers (Ries 2015). The mechanical characteristic values of the material as a consolidated SRC are given with a tensile strength of 200 MPa, an elastic modulus of 5500 MPa and a breaking elongation of 9%.

The injection molding component used was PP 520P from Sabic (Sabic Deutschland GmbH & Co. KG, Gelsenkirchen, Germany) in the form of plastics granulate. It has an MFR of 10.5 dg/min (at 230 °C and 2.16 kg), a density of 0.905 g/cm³ and a tensile strength of 36 MPa. The stiffness of the material is given as 1700 MPa. The material offers a wide temperature range from 180 to 280 °C in which it can be processed.

2.2 Processing

The tool used is a compacting and injection molding tool specially developed for the combined compaction and back-injection molding process. It has heated press plates with an overall heating capacity of 25.6 kW and water cooling. The tool enables a heating rate of approx. 10 °C/min and a cooling rate of up to 100 °C/min. A more detailed description of the tool can be found in Jakob et al. (2021). The cavity for back-molding is positioned on the gate side and is shown with a cross-section of 20 × 4 mm² by way of example in Figure 1.

This tool has been specially developed for the combined compaction and back-molding process and has three temperature and pressure sensors in the cavity. This enables the tool wall temperature and the cavity pressure, referred to as local variables below, to be recorded at three points over the flow path. The precise influence of the machine parameters on the local variables has already been set out in Jakob et al. (2021).

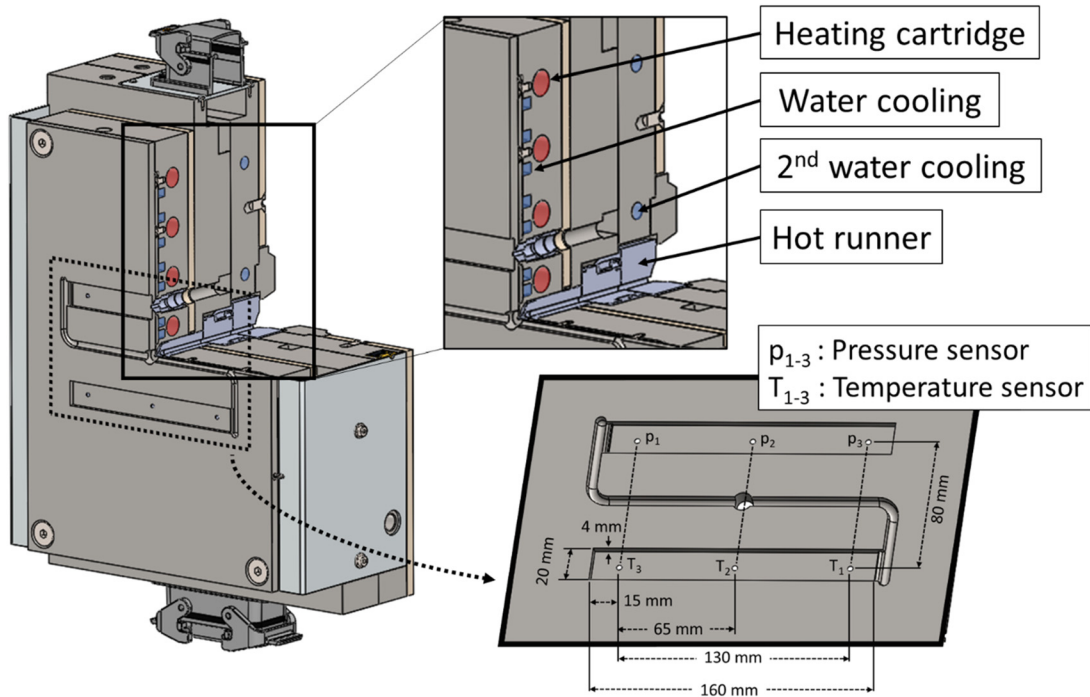


Figure 1: Tool for the combined compaction and back-injection process (Jakob et al. 2021).

For the tests, use was made of a cavity with a cross-section of 4 mm in width and 20 mm in height. Thus, the shape of the cavity is similar to the form of a stiffening rib. The back-molded composites were subsequently loaded to failure in a shear-off test and a pull-off test.

Six layers of PURE[®] fabric were used to make the SRC-PP composites and these were hung inside the ready-heated mold. Immediately prior to the start of the process, the melt is metered. Once the fabric stack has been hung in the tool, the tool is closed, and the compaction pressure built up. Consolidation is performed with a pressure of 7 N/mm² and a holding time of 60 s. For the tests, the barrel wall temperature (T_b) was varied between 180 and 280 °C, the time of Injection (t_i) between -30 and 30 s and the tool temperature between 130 and 150 °C. The temperature range of the tool was deliberately kept low in order to consolidate the SRCs, but to keep the influence of the tool temperature on the orientation as low as possible. Three specimens were removed over the length of the flow path for each parameter setting, and a total of 456 test specimens were tested in this way. These are summarised in Table 1.

The time of injection set out in the test plans is expressed in terms of the start of cooling and can thus assume negative values. For an easier understanding, Figure 2 shows the pressure and temperature curves over time.

2.3 Preparation

Specimen preparation was performed with a CNC controlled specimen milling machine from Coesfeld (Dortmund, Germany). The specimens with a length of 70 mm were removed in the area of the sensors, orthogonal to the direction of flow in each case, and milled to a parallel specimen width of 18 mm. This results in a shear surface of 72 mm² through the width of the overmolded specimens. The precise position of specimen removal from the composites and the dimensions of the specimens are shown in Figure 3. By removing the specimens at different positions, it is also possible to assess the influence of the distance from the gate (D_G).

Table 1: Experimental setup for shear-off and pull-off tests.

Specimens	Test setting	Parameter	Unit	Setting levels				
456	228	Shear-off test	Barrel temperature	[°C]	180	230	280	
			Time of injection	[s]	-30	-10	0	10
	228	Pull-off test	Tool temperature	[°C]	130	140	150	

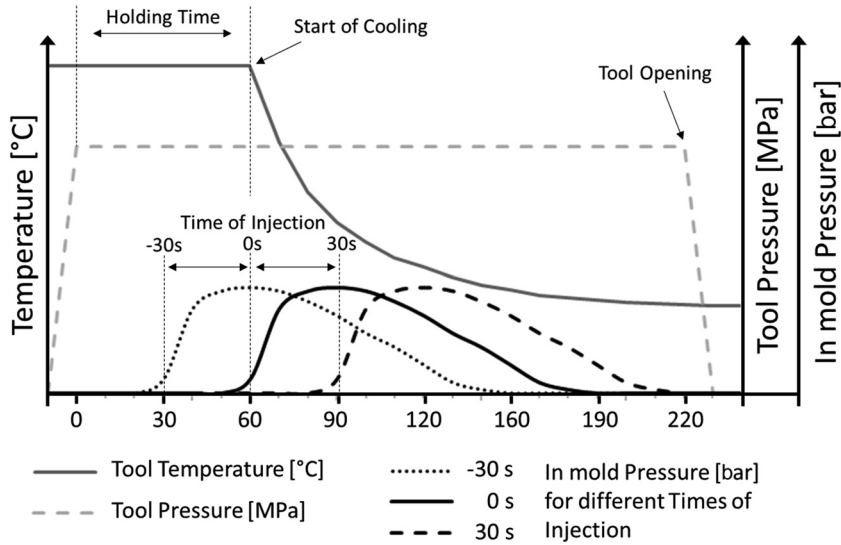


Figure 2: Schematic illustration of temperature and pressure over time for the combined compaction and back-injection process.

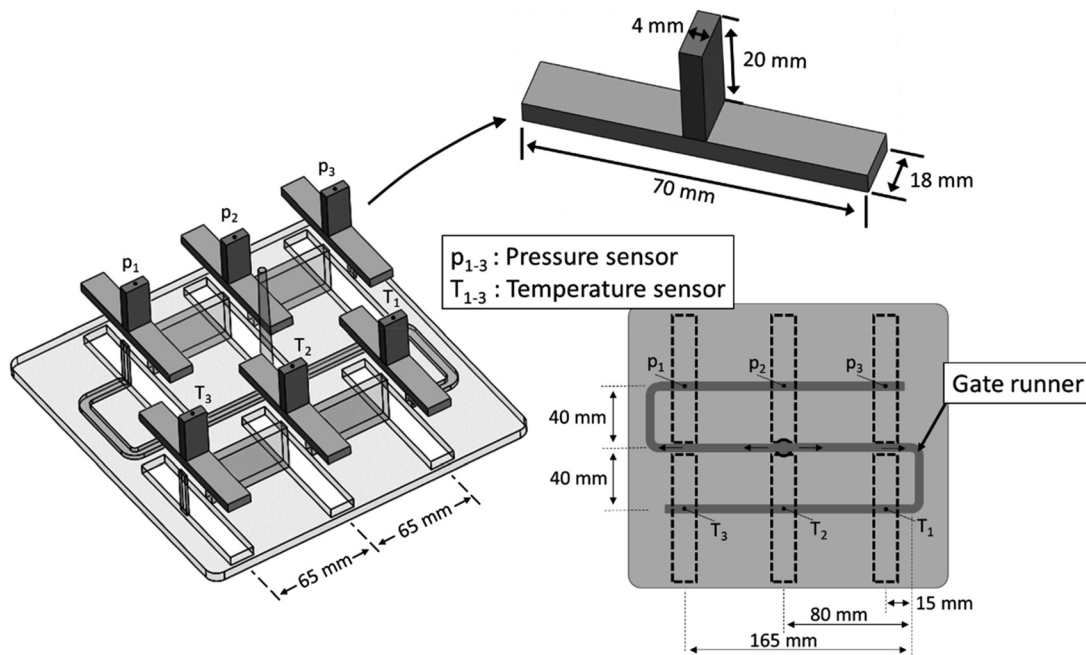


Figure 3: Preparation position of the test specimen (2D and 3D) and test specimen geometry for the shear-off test.

2.4 Measuring

The shear-off tests are conducted on a universal tensile and compression testing machine from Zwick Roell (Ulm, Germany). Use was made of a 10 kN force measurement sensor and a testing device as shown in Figure 4A. The test velocity is specified at 10 mm/min.

For the evaluation, the maximum shear stress in the shear surface was determined on 228 specimens. The stiffness and maximum elongation at break were not determined, since these are not expected to permit any statement to be made regarding adhesion. The device

shown in Figure 4A has a gap that can be adjusted between the rear guide surface and the shear-off edge. It is thus possible to achieve a setting that permits good guidance of the SRC without the specimens getting clamped. To prevent the injection molded component from twisting, this is additionally fixed from underneath.

The pull-off tests are conducted by pulling off the rib orthogonally to the SRC on 228 specimens. To do this, the specimens are placed in the device shown in Figure 4B and are fixed in place by a clamping bar. The injection molded body is then clamped under the device in grips and the entire device pulled off upwards.

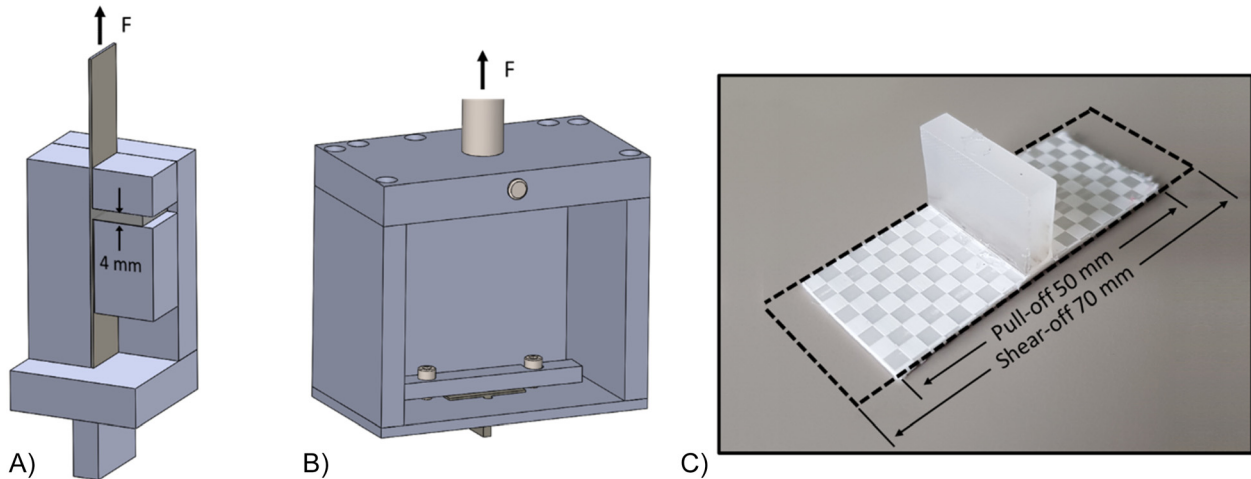


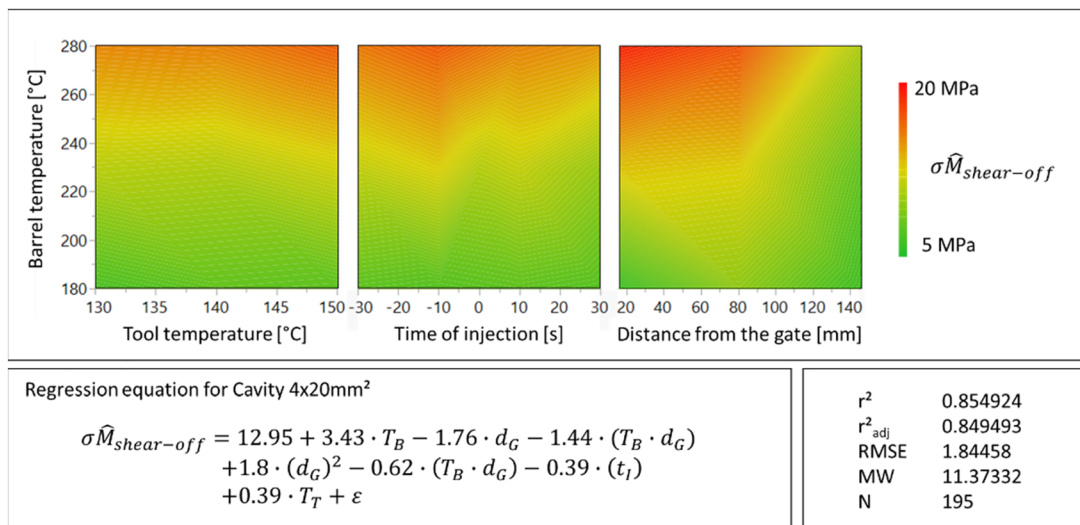
Figure 4: Testing device for (A) shear-off tests, (B) pull-off tests and (C) picture of a specimen.

3 Results and discussion

3.1 Shear-off tests

The evaluation is performed with the aid of regression models compiled with JMP Pro, Version 15.0.0 (SAS Institute GmbH, Heidelberg, Germany). The procedure for compiling the model is described in Jakob et al. (2021). The results of the regression model for the maximum shear stress ($\sigma \hat{M}_{shear-off}$) are shown in Figure 5. The regression equation set out there was created with standardised target variables to ensure comparability of the influencing factors.

On the basis of the regression equation, it is clear that the barrel temperature has the greatest influence on the maximum shear stress. The shear-off strength increases with barrel temperature from 8.87 MPa at 180 °C to 17.3 MPa at 280 °C. The mobility of macromolecules increases with rising temperature, this leads to better interdiffusion at the interface and thus to a better adhesion (Ehrenstein 1999). An increase in the mold temperature also has a positive influence on the shear-off strength, leading to a rise from 12.63 MPa at 130 °C to 13.54 MPa at 150 °C. With a later time of injection, there is similarly a reduction in the shear-off strength from 13.66 MPa at -30 s to 12.51 MPa at 30 s. A clear



T_B = Barrel temperature [°C]

t_i = Time of injection [s]

d_G = Distance from the gate [mm]

T_T = Tool temperature [°C]

ε = Model error [-]

Figure 5: Results of regression modelling for the shear-off strength of a back-injected specimen.

reduction in the shear-off strength is seen with an increasing distance from the gate, from 12.76 MPa at 15 mm to 8.01 MPa at 145 mm. This data applies with the factors that are not mentioned being fixed at an average setting within the parameter range. The model values are between 4.37 and 21.15 MPa in overall terms.

3.2 Pull-off tests

The pull-off tests are similarly evaluated with the aid of JMP Pro, Version 15.0.0. The approach adopted for drawing up the regression models was the same as that adopted for drawing up the regression models for the shear-off tests, and the presentation of the results is also the same as previously. Figure 6 shows the pull-off strength curve as a function of the parameters.

It is clear that the maximum pull-off strength determined also increases as the barrel temperature rises. With a barrel temperature of 180 °C, the pull-off strength is 3.39 MPa, rising to 5.42 MPa for a barrel temperature of 280 °C. The influence of the tool temperature cannot be clearly defined. The results show a maximum pull-off strength of 4.41 MPa at a tool temperature of 150 °C. The minimum value determined in the regression model is 4.08 MPa at 138.5 °C. The influence of the time of injection is clear to see. Here, the pull-off strength falls from 4.46 MPa with a time of injection of –30 s to 3.69 MPa for a later time of injection of 30 s. The influence of the distance from the gate shows a maximum pull-off strength of 4.11 MPa with a

distance from the gate of 67.5 mm and a minimum pull-off strength of 2.99 MPa with a distance from the gate of 145 mm. This data applies with the factors that are not mentioned being fixed at an average setting within the parameter range. Overall, the model values are between 1.92 and 6.13 MPa. By comparison, the average pull-off strength, at 4.06 MPa, is 7.61 MPa lower than the shear-off strength at 11.67 MPa.

3.3 Microscopic observations

The clear difference in the maximum stress recorded in the two test methods suggests that the type of loading has an influence on the result, or that failure does not occur in the joint plane. On the basis of the microstructures visible in the cross-section of the composites under the microscope, a potential failure case is shown in the form of a model in Figure 7. The microscopic images have been taken with the aid of a confocal laser light microscope. The etching method used for preparing the specimens, which exposes the microstructures of the composites, is described elsewhere (Ehrenstein 2019; Ries 2021).

The tapes of the fabrics used have been produced in a multilayer film extrusion process with the application of a lower-melting edge layer. The melting temperature of this edge layer is between 130 and 140 °C, which is why it can be assumed that it will melt sufficiently with the melt injected at a temperature between 180 and 280 °C, thus achieving good adhesion. The better the bond between the injection-

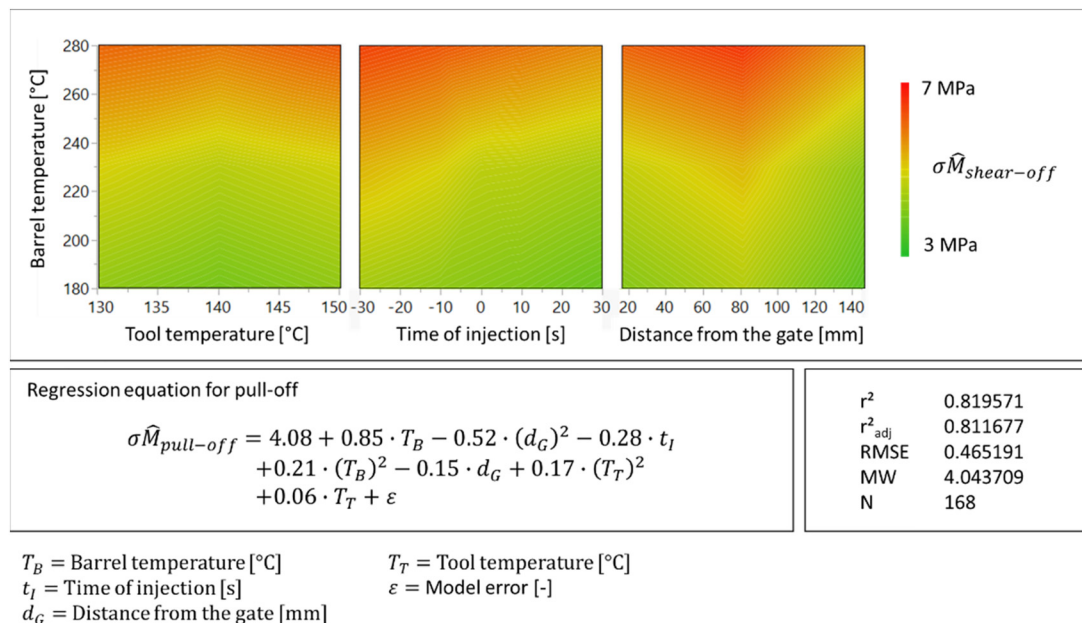


Figure 6: Results of regression modelling for the pull-off strength.

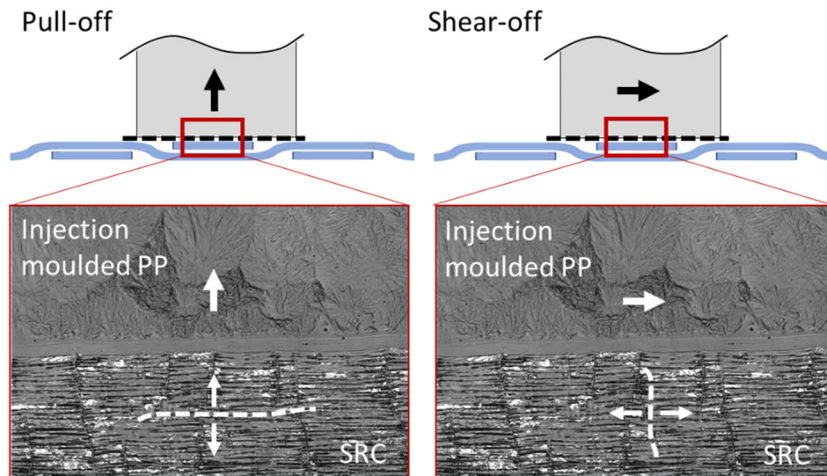


Figure 7: Schematic illustration of the failure mechanism in SRC as a function of the test mode.

molded component and the SRC, the better the force can be transmitted from the injection molded component to the SRC. In the pull-off tests, this means that the SRCs are loaded transversely to the orientation in the tapes. It is known from various publications that, with orientation in a preferred direction, the strength transverse to this direction is considerably reduced (Alcock 2004; Ehrenstein 1999; Schimanski 2002; Ward and Hine 2004). With a comparable material system, it was shown that failure in a peel test always occurs within the tape if the compaction temperature is high enough (Alcock 2004). The maximum peel strength achieved with the tapes used is given as 1.2 N/m at a compaction temperature of 180 °C. Shear-off tests were additionally performed in which a strength of some 27.5 N/m was seen for a compaction temperature of 160 °C, as a function of the shearing surface. This almost matches the strength of the tapes used (30 N/m).

The loading types can be readily transposed to the tests conducted here on the compacted and back-molded SRCs. While the pull-off test leads to the SRC being loaded transversely to the fibre orientation, the force in the shear-off tests is introduced in the direction of orientation.

In order to investigate the failure in more detail, scanning electron microscope images were taken of the fracture surfaces on the SRCs. After mechanical testing, the injection molded parts still adhered to the SRC in some areas. These have to be carefully removed for recording the images. Figure 8 shows the fracture surfaces obtained with the different test methods for two selected process points. The differences between the selected process points are clearly visible from the damage. For the shear-off tests, the shear direction is indicated by an arrow in each case.

In the images of the fracture surfaces of the parts tested in the shear-off test, it is clear that the tapes transverse to the loading direction, in particular, are damaged to different degrees as a function of the parameters. With a barrel temperature of 280 °C and a time of injection of –30 s, the transverse tapes are almost completely torn off. A fracture can be seen in the tapes running in the load direction, combined with a kind of folding of the tapes. The tapes seem to be more compact, i.e. less fibrillated, than the fracture surface of the shear-off samples at a barrel temperature of 180 °C and a time of injection of +30 s. In addition, some tapes in the direction of the shear-stress have not failed. For those tapes that have failed, microfibrillation of the tapes has occurred, which points to a lower transverse strength. Detailed images of the fracture surface are shown in Figure 9.

The pull-off tests similarly show a clearly dissimilar fracture surface as a function of the parameters. With a barrel temperature of 180 °C and a time of injection of +30 s, it is clear that failure takes place primarily adhesively with only isolated splicing into microfibrils. By contrast to this, a barrel temperature of 280 °C and a time of injection of –30 s leads to clear splicing and also to a clear failure within the tapes. The detailed images are shown in Figure 10.

A comparison of the two test methods shows that, with the pull-off method, the tape lifts off the SRC. This is particularly evident in Figure 8C, where the tape forms an arc in the direction of the pulled-off injection molded part. The fracture surface consequently shows that there is also a peeling effect when the specimens are pulled off, while in the fracture surfaces for the shear-off test, everything points to the introduction of force into the tapes.

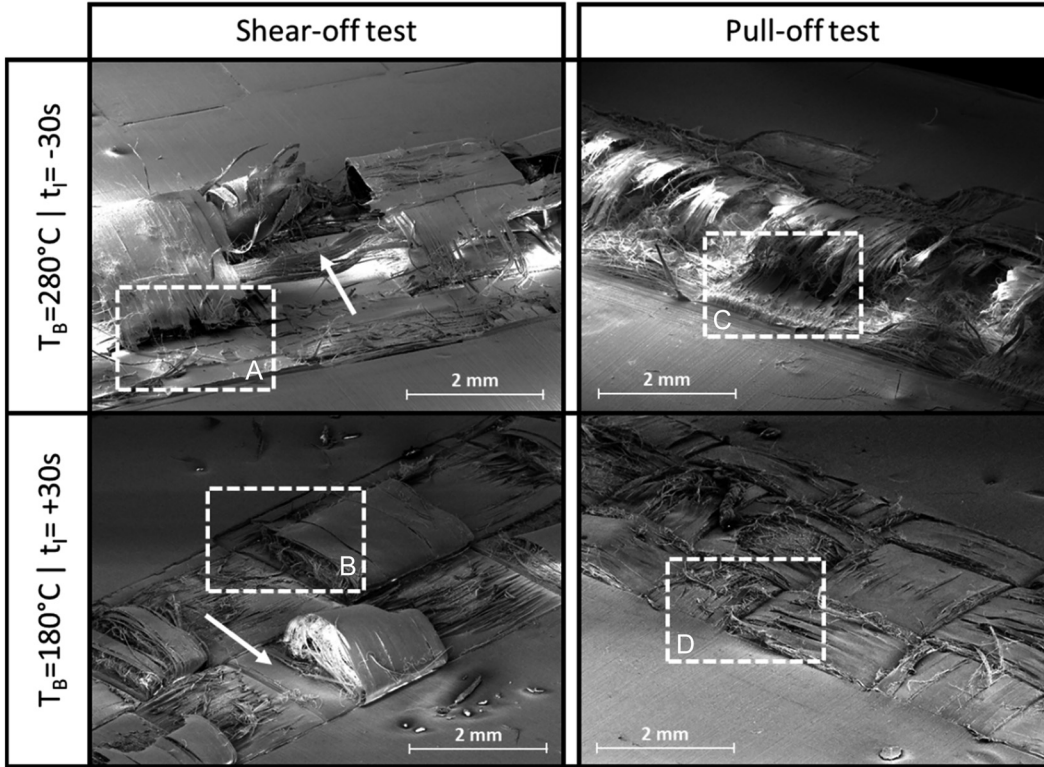


Figure 8: SEM pictures of the fracture surface for different test methods as a function of the process parameters.

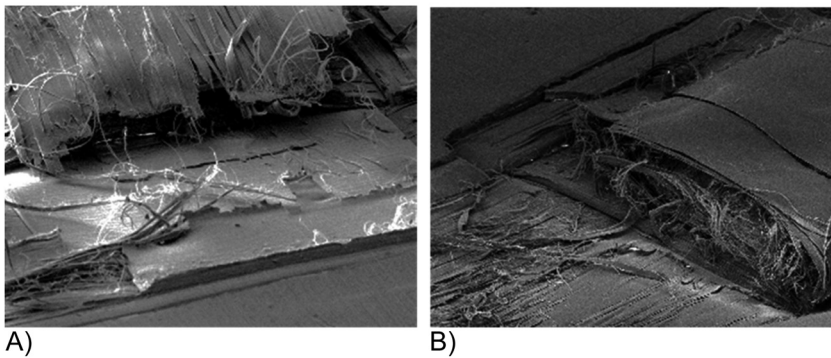


Figure 9: Detailed SEM images of the fracture surface from Figure 8A fracture of the tape and from Figure 8B splicing into microfibrils and folding of the tape.

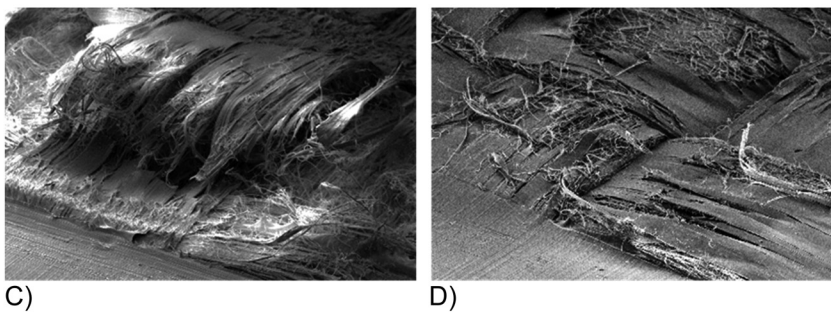


Figure 10: Detailed SEM images of a fracture surface from Figure 8C splicing into microfibrils and from Figure 8D surface fracture.

4 Conclusions

The adhesion of the injection molded component to the SRCs can be improved by greater consolidation, achieved through a higher temperature of the melt and the mold and through an earlier time of injection during the process. Since it is well known that the chain mobility increases with rising temperature and this leads to better interdiffusion at the interface, this effect was to be expected. From the microscopic images, it can be seen that, initially, the adhesion between the tapes and the injection molded plastic is decisive for the adhesion, which improves as the contact temperature rises. At the same time, the probability of failure within the tapes rises with greater adhesion in the interface. The tapes have only a low transverse strength, but this can be enhanced by increasing the orientation of the macromolecules in the transverse direction (Alcock 2004; Schimanski 2002). The increased transverse strength goes hand in hand with a reduction of the orientation in the longitudinal direction and hence with a reduction in the mechanical strength of the SRC.

Under loading in a shear-off test, an optimum process setting exists that permits good force introduction into the tapes through a sufficient level of adhesion. To determine the optimum process, point for the production of back-molded SRCs, it is necessary to take a holistic view of the SRCs and also make allowance for the strength and stiffness of the SRCs, for example.

On the basis of the available findings, it will, in future, be possible to make allowance for the strength, which is a function of the type of force introduction, at the component design stage. The distance to the gate can similarly be taken into account as a decisive factor for adhesion, and provision can be made for corresponding hot runner systems in the planning of molds.

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