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Characterization of Anisotropic Properties of Hot Compacted Self-Reinforced Composites (SRCs) via Thermal Diffusivity Measurement

The mechanical properties of self-reinforced composites (SRCs) produced in a hot compaction process significantly depend on the process parameters. Only a little deviation of the process temperature or pressure causes the component to act differently under mechanical load. This is a chance and a challenge at the same time, since this process is difficult to handle but by properly controlling the process parameters, the mechanical properties can be adjusted, even locally for one component. In this research SRC are manufactured in a hot compaction process. A correlation between process parameters and density is found. Density increased from 0,8 to 0,91 g/cm³ by increasing temperature and pressure in the hot compaction process. The different thermal properties in the direction of orientation (IP) and transverse to orientation (TP) are measured with a laser flash device. It was found that, due to a change in density and molecular orientation, diffusivity and conductivity are influenced in different degrees in IP and TP directions. For interpretation of thermal measurement results, microstructures are analysed with a confocal laser scanning microscope after preparing the specimen with a permanganate etching. A schematic model of conductive path is worked out and discussed. With measurement data the anisotropy of IP and TP diffusivity is calculated, and a model is built to describe relative density related to anisotropy. The highest anisotropy between IP and TP diffusivity was calculated with a ratio of 6 at a relative density of approximately 0,82 g/cm³. Since mechanical properties in correlation to process parameters have already been investigated, results of this investigation, in combination with previous research on mechanical properties, will enable the development of a non-destructive testing method for SRCs by measuring the thermal diffusivity.

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1 Introduction

In contrast to conventional composites, the fibres and matrix of self-reinforced composites (SRC) are based on the same type of polymer. The advantages of SRCs are their recyclability, a high stiffness, good tensile strength and, in case of special polymers such as polypropylene, their outstanding impact resistance. Self-reinforced composites can also be used in lightweight applications; for example, some special SRCs made of PP reach a density of 0.78 g/cm² (PURE, technical data sheet, DIT B.V., Dinxperlo, The Netherlands, 2016). For this reason, SRCs are often used in sports equipment and the automobile industry.

However, there are some challenges that are associated with using this type of material. Due to the complex manufacturing process, SRCs are expensive. Additionally, the processing parameters must be controlled very carefully. The mechanical properties depend on the processing parameters to a large extent. This represents an opportunity and a challenge at the same time. The mechanical properties can be adjusted in a wide range by changing process parameters and therefore easily be customized for the needs of the application. On the other hand, the requirements for process continuity and process control are very strict. Only a few degrees deviation in the processing temperature can lead to insufficient component quality.

Until 2010, research mainly focused on polypropylene, partly due to its good impact properties in SRCs. In recent years, some research groups have focused on processing SRCs based on PET (Schneider et al., 2013; Jerpdal and Åkermo, 2014; Jerpdal et al., 2016; Poulidikou et al., 2016; Jerpdal et al., 2018; Zhang and Peijs, 2010; Chen, 2011; Andrzejewski et al., 2016). Additional current focuses of research include hybridisation with carbon fibres from SRCs based on PA (Hine et al., 2014; 2017) and PP (Selezneva et al., 2018; Mesquita et al., 2018; Tang et al., 2018) as well as hybridisation with steel fibres (Swolfs et al., 2017).

Other researchers have been concentrating on process-initiated grading of SRCs (Bledzki et al., 2008; Paßmann, 2009; Heim et al., 2013a; Ries, 2015). Grading means the processing sensitivity is used to specifically adjust the mechanical properties of different areas of a component to produce varying mate-

rial properties within the same component while using the same raw material. Since the processing parameters pressure and temperature differ locally, the material properties can be varied strongly within a component, making it possible to incorporate a broad spectrum of strength, stiffness and impact properties. This is especially true for SRCs based on PP (Ries, 2015).

None of the mentioned studies focused on the thermal conductivity or thermal diffusivity of SRCs. Particularly in the case of graded SRCs, the ability to control these properties after processing would be of great benefit.

As the mechanical properties have already been investigated in several studies, this paper focuses on describing the microstructure in SRCs by measuring their thermal diffusivity. As already known, the mechanical properties of SRCs based on PP strongly depend on the microstructure (Bledzki et al., 2012; Ries, 2015; Homberg et al., 2013; Heim et al., 2013b). The correlation between these microstructures and the mechanical properties has been the subject of numerous studies (Ries 2015; Heim et al., 2013a; Paßmann 2009; Bledzki et al., 2012). This paper presents new findings for the thermal properties in correlation with the microstructures of SRCs. These new findings will make it possible to draw conclusions about the mechanical properties of SRCs when measuring their thermal diffusivity. The presented results could be used for the development of a non-destructive testing method for SRCs in the future.

2 State of Technology

Polymers do not consist of ideal crystals, but instead of chain molecules arranged in structures that can be more or less regular. Pietralla (1981) describes that heat transport in the direction of the chain of macromolecules is a one-dimensional process. The most important form of heat conduction inside a solid, unfilled polymer is energy transport by lattice vibrations (phonons). Based on the theory of the scattering of phonons at interfaces, it is assumed that only the transport within a molecular chain can be regarded as an essential transport mechanism for heat. For this reason, the main valence bond is assigned a thermal resistance ten times lower than the secondary valence bond (Pietralla, 1981).

In self-reinforced polymers, this results in a much higher thermal conductivity in the direction of orientation than transverse to it, since the transport transverse to the molecule chains is mainly characterised by the weak interactions of the secondary valence bonds. This measurable anisotropy can be observed in all polymers when the molecular chains are aligned parallelly in a specific direction, and this is applicable regardless of whether a polymer is partially crystalline or amorphous. Thus, a higher orientation leads to a larger anisotropy of the thermal conductivity (Hellmuth et al., 1967; Kurabayashi, 2001; Novichenok and Ovchinnikova, 1982).

Various studies exist on the anisotropy of thermal conductivity in stretched materials. Hellwege et al. (1963) carried out measurements on various amorphous samples stretched in a tensile testing machine. The maximum anisotropy values achieved were 3 for amorphous polymers (Hellmuth et al., 1967; Hellwege et al., 1963) and 7 for semi-crystalline poly-

mers (Kurabayashi and Goodson, 1999). Based on theoretical calculations, values of 10 to 30 are possible (Hellmuth et al., 1967). An overview of existing studies is provided in Kurabayashi (2001).

Recently, this topic was discussed for special materials. Bai et al., showed that the relative thermal conductivity of a PLLA film (0.196 W/mK) increases to 0.348 W/mK in the in-plane direction while it decreases to 0.125 W/mK in the through-plane direction at a tensile strain of 4 (Bai et al., 2018). Zhu et al. (2017), found a significant enhancement of the thermal conductivity in a UHMW-PE microfibre by stretching it and applying heat. They measured an increase of the thermal conductivity from 19–22 W/mK to 50.8 W/mK.

These studies mentioned here focus on thermal conductivity in oriented foils and fibres, especially thermal conductivity enhancement when stretching polymers subject to elevated temperatures. In contrast to the mentioned researches, this study does not examine the improvement of the thermal conduction in the direction of orientation. It focuses on the reduction of thermal diffusivity in the direction of the polymer chain orientation in SRCs as a result of changes to processing parameters in a hot compaction process.

Other studies on thermal diffusivity and the conductivity of conventional fibre-reinforced composites with non-thermoplastic fibres like glass, carbon or any natural fibre are not relevant for this study. These fibres do not have a temperature-dependent microstructure that changes in connection with processing parameters. Thus, in conventional fibre-reinforced composites no change in the thermal properties of the fibres occurs as a function of the processing parameters like in SRCs.

3 Experimental Studies

3.1 Materials

A fabric named PURE made by DIT B.V., Dinxperlo, The Netherlands, was used for manufacturing the test samples. This is a highly oriented tape made of 100% polypropylene that was specially developed for hot compaction.

The individual tapes are 2.2 mm wide and approx. 0.07 mm thick. Their stiffness is 14 GPa and their tensile strength 500 MPa (PURE, technical data sheet, DIT B.V., 2016). The tapes are made of three layers; the outer layers have a low melting point and allow good adhesion of the individual tapes in the hot compaction process. When used in hot compaction, the individual tapes are woven into a plain weave fabric with an area weight of 0.105 kg/m² (PURE, technical data sheet, DIT B.V., 2016). The measured density of the tapes is approximately 0.809 g/cm³, which is 12% below the density of polypropylene (approx. 0.92 g/cm³). This reduction in density can be explained by defects within the tapes caused by the deformation processes (Alcock 2004). These defects, as well as the reduced density, occur at stretching ratios above 1:9 and are the result of the beginning of fibrillation within the formation of microvoids (Alcock, 2004). The materials were processed into composite panels with a thickness of at least 10 mm.

3.2 Processing

The composites were produced in a hot compaction process on a hydraulic thermal press (model Joos LAP 80, Gottfried Joos Maschinenfabrik GmbH & Co. KG, Pfalzgrafenweiler, Germany). 94 layers of fabric were stacked to produce a composite panel that was at least 10 mm thick. The layers were continuously cut from one fabric roll. Stacking was achieved by turning each layer 90° to make sure that the SRCs were stacked to maintain the planar isotropy. The manufacturing was done by varying the processing temperature and the compaction pressure. The temperature was measured in the core of the panels using a temperature sensor which was placed between layers 47 and 48. The heating rate was kept constant while the processing time increases at higher compaction temperatures.

The aim of the experimental design was to vary the macromolecular orientation in the composites. It was assumed that the orientation of the macromolecules in the tapes used is maximum and a complete melting, relaxation and recrystallization of these is equivalent to the minimum orientation. To achieve this, the parameters temperature and pressure were varied over several stages. The temperature was adjusted within the range 165 to 190°C, the compacting pressure was adjusted in the range 1.6 N/mm² to 3.6 N/mm². Figure 1 shows the exact parameters of each SRC processed for this paper.

3.3 Preparation

First, 100 × 100 mm composite parts were cut out of the centre of each SRC (12 plates with different parameters as shown in Fig. 1). The cut borders in all directions were at least 50 mm wide. All samples used for testing were cut out of these 100 × 100 mm plates with a distance of at least 50 mm from the centre.

The final test samples were prepared by cutting. In accordance with DIN EN ISO 2818, a saw blade with a diameter of 80 mm, 50 teeth and a thickness of 1 mm was selected for cutting. In order to prevent additional heat input into the material, the material and the saw blade were cooled using a compressed air driven heat exchanger. An infrared temperature measuring device was employed during the cutting operation to measure

any heating and temperatures up to a maximum of 30°C were determined. 10 × 10 × 2 mm samples were used for the thermal conductivity measurements which were recorded as the comparative measurements in the direction of orientation (IP) and transverse to the orientation (TP), as shown in Fig. 2.

Six test samples were taken from each plate for density measurements. In the case of the TP samples, it was ensured that equal parts of the samples were taken from the surface layer of the composite panel and the centre. 15 samples were taken from each panel for the thermal diffusivity measurements. In each case, 9 IP samples and 6 TP samples were prepared.

3.4 Measuring

The thermal diffusivity (a) was measured using a laser flash device (model LFA 467) manufactured by Netzsch, Selb, Germany. The measured area was a spot 5 mm in diameter in the centre of each sample. In order to calculate the thermal conductivity (λ) based on Eq. 1, the specific thermal capacity (c_p) and the density (ρ) of the material are required additionally. The former was determined by means of DSC analysis (model DSC Q1000, TA Instruments, New Castle, USA). The density of the samples was determined using the immersion method according to ISO 1183-1. Due to the low density of the samples (below 1 g/cm³), isopropyl was used as liquid for testing.

$$\lambda = a * \rho * c_p. \quad (1)$$

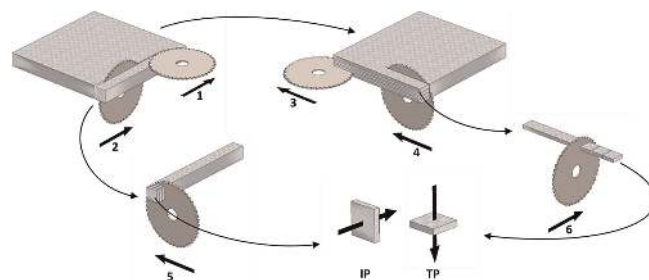


Fig. 2. Preparation procedure of samples for thermal diffusivity measurement

Nr.	Pressure	Temperature
1	1.6 MPa	165°C
2		169°C
3		178°C
4	2.2 MPa	165°C
5		169°C
6		174°C
7		180°C
8		181°C
9	3.6 MPa	165°C
10		179°C
11		187°C
12		190°C

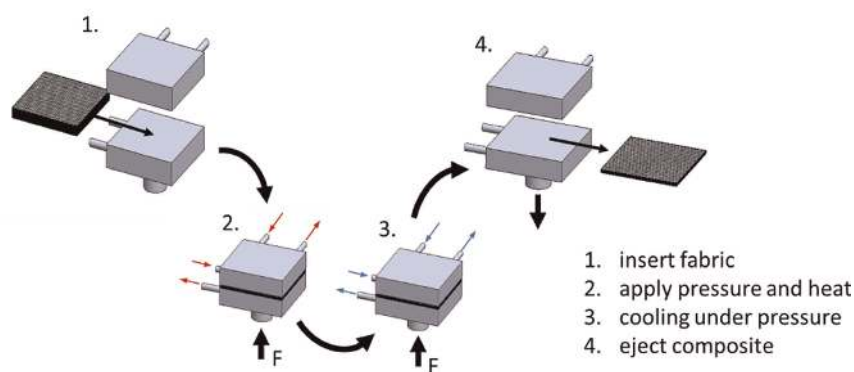


Fig. 1. Sets of processing parameters for manufacturing each SRC and schematic illustration of the hot compaction process

The density was measured on two types of samples. The first were cubes with a length, width and height of 10 mm, which represent the density of an SRC plate compacted with a certain set of parameters. The second were platelets ($10 \times 10 \times 2$ mm) that were used for thermal diffusivity measurements to be able to assign a density value to every thermal diffusivity value.

4 Results

4.1 Density

The measured density of the composite panels shows that the density increases as the pressure and temperature increase. Figure 3 shows the influence of the temperature on the density at different pressure levels.

In general, the measured density of the composites approaches the density level of injection molded polypropylene as the temperature and pressure increase. The influence of the temperature on the density is greater than the influence of the compacting pressure.

As already mentioned in Section 3.1, the density of the tapes used was lower than that of polypropylene due to the microvoids produced by the drawing process (Alcock, 2004; PURE, technical data sheet, DIT B.V., 2016). As a result of heating the material and applying pressure, these cavities closed again, which explains the increase in density in correlation with the processing parameters. This also means that the closure of the microvoids is linked to a reduction in orientation. An increase in density can therefore also be indirectly associated with a reduction in orientation. However, there are other local parameters that influence the density and they are discussed below.

Owing to the structure of the fabric, air became enclosed between the tapes. This especially occurred in the crossover areas. The parallel orientation of macromolecules causes the material to have crystal-like structures. The orientation creates a similar high packing density of the polymer chains, whereby, as a result of the atomic distance, similar strong secondary valence forces are formed like in crystalline areas (Ehrenstein, 1999). If these are melted, the recrystallization will lead to the formation of conventional spherulitic crystalline structures.

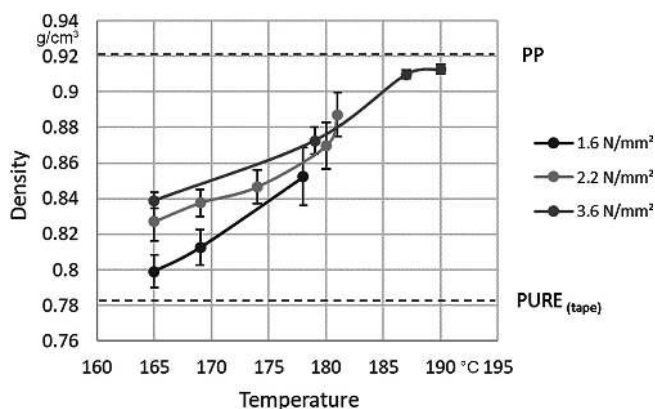


Fig. 3. Influence of processing parameters on the density

The change in the degree of crystallisation is therefore minute, since a change or rearrangement of the crystal structure is more likely than a measurable change in the quantitative crystallinity. Due to this effect, the influence of the crystallinity on the density is rather minor compared to the previously mentioned air enclosures.

The processing parameters significantly influence the extent of matrix in the composites, the degree of orientation of the macromolecules in the individual tapes, as well as the quantity and size of the microvoids. As the variations in the measured density show, there is a correlation between the processing parameters and the composite density. Since density is a locally measured quantity that can be determined unaffected by non-adjustable variables during the manufacturing process, it was used as the basis for presenting the results in the following section.

4.2 Microscopy

The difference in the density of the composites can be seen in the structural characterisation. The samples were ground first, polished, and then treated with chemical etching to expose their structure (Olley et al., 1979; Breiling et al., 1997; Ries, 2015; Heim et al., 2013a). Figure 4 shows the structure of the tapes in the IP and TP directions. For the purpose of better illustration, the structure within the tapes is shown here as a 3D model created from 2D images. Moreover, Fig. 4 shows the structure that can be assigned to the different areas in the tapes. Figure 5 shows areas of different manufactured composites in which both the IP and TP structures are visible to make it easier to understand the change that occurs due to the processing parameters

There are fibril bundles inside the lower tape which are held together by a few crossbands. The microvoids within the tapes are also clearly visible. The tape at the top of the image shows the structure of the transverse section. In this direction, the microvoids look like a kind of net. The coextruded layer on the tapes can also be seen between the tapes. The changes in the structure caused by the processing parameters are shown in Fig. 6.

As the compaction temperature increases, the microvoids close more. This is clearly evident when comparing sections a and c in the images, see Fig. 7. In the upper sections of the images, the microvoids are dark areas in the transverse area which become increasingly smaller as the processing tempera-

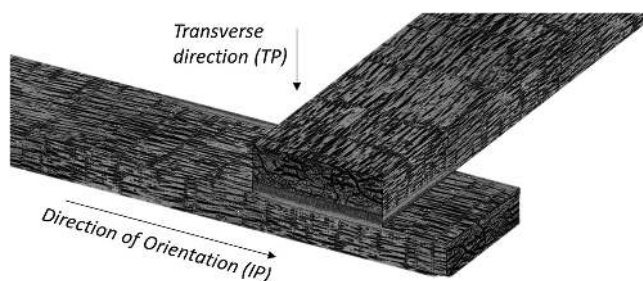


Fig. 4. Qualitative three-dimensional representation of two crossing tapes with exposed inner structures (no scale)

ture rises. The oriented structures are visible throughout the entire parameter range and are not completely melted. However, the influence on the orientation of the individual macromolecules can only be assumed based on the microscopic images.

4.3 Thermal Properties

Figure 8 shows the relative change in the IP and TP thermal diffusivity in relation to the density. The absolute change in diffusivity within the measured density range from 0.8 to 0.92 g/cm³ is 0.05 [mm²/s] for IP and 0.0367 [mm²/s] for TP. While taking the mean measured diffusivity (IP = 0.43 mm²/s; TP = 0.08 mm²/s) into consideration, this results in a change in the IP diffusivity of approximately 12% and approximately

45% for the TP diffusivity. Figure 9 shows the influences on the thermal conductivity. In the TP direction, the thermal conductivity increases by 58%, which is even more than the diffusivity in the range 0.8 to 0.92 g/cm³. In contrast, the IP values even show a small increase towards higher densities.

5 Discussion

For the following discussion, it is important to highlight the difference between diffusivity and conductivity.

- Thermal diffusivity describes how fast a thermal impulse can move through a material – this parameter reaches high values when there is an almost direct conduction path in the material.

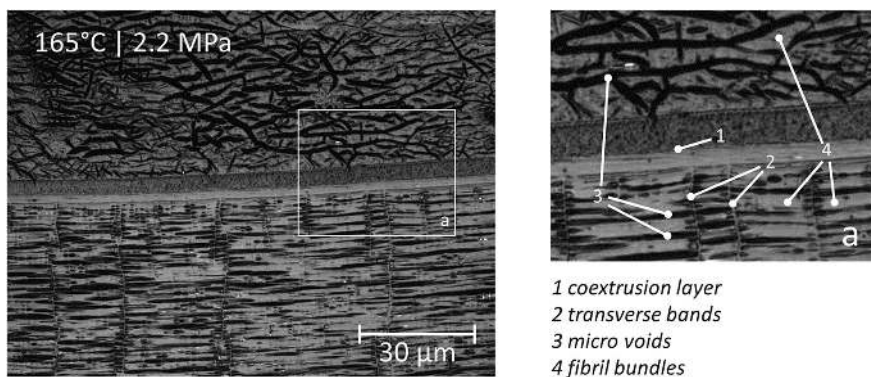


Fig. 5. Laser light microscope image of a composite treated by chemical etching, compacted at 165°C/2.2 MPa

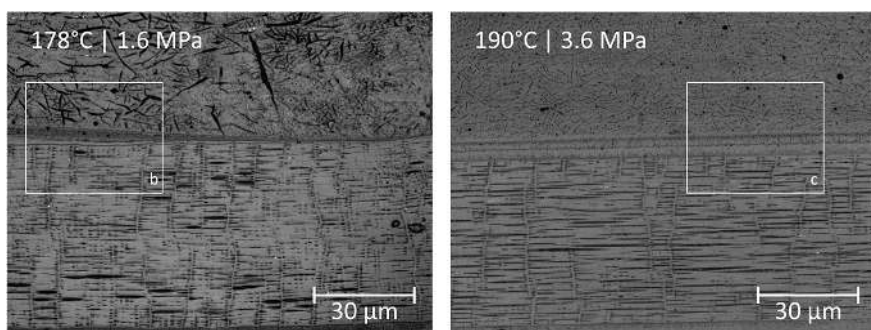


Fig. 6. Laser light microscope image of a composite treated by chemical etching, compacted at 178°C/1.6 MPa (left) and 190°C/3.6 MPa (right)

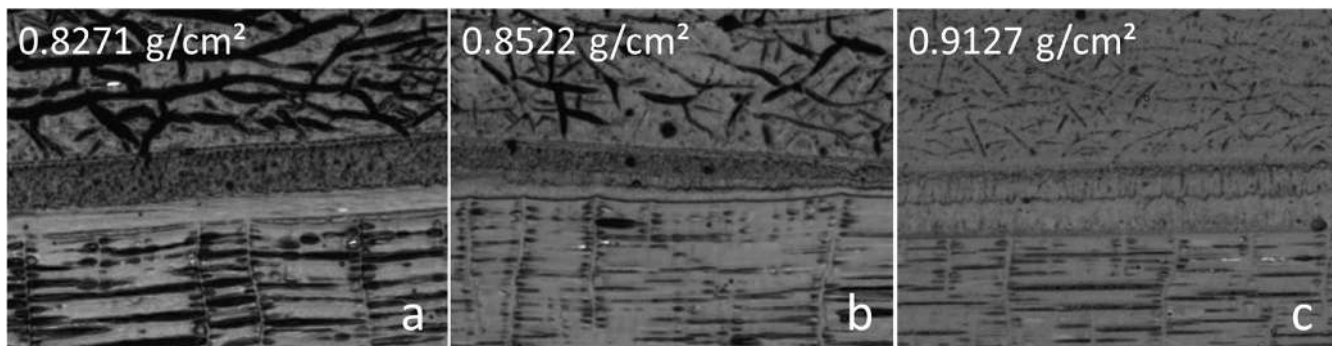


Fig. 7. Sections a (165°C/2.2 MPa), b (178°C/1.6 MPa), and c (190°C/3.6 MPa) of the microscopically examined composites

- Thermal conductivity describes the amount of energy that is transported through a sample over a certain period at a defined temperature difference – the effective cross-sectional area as well as the specific heat capacity of the material are included in this parameter.

The results have shown that the increasing density resulting from a higher degree of compaction is associated with a change in thermal diffusivity and conductivity. In percentage values, the TP properties are more strongly influenced than the IP properties. Two factors are important here. First, as the degree of melting increases, the orientation of the molecules decreases. Second, air enclosures between the tapes and micro-

voids within the tapes (see Fig. 7) reduce as the temperature and pressure increase.

These two factors have different effects on the IP and TP diffusivity. The orientation of the macromolecule chains can be considered equivalent to the orientation of the covalent bonds. This results in an elongation of the conduction path along the covalent bonds in the IP direction, because the molecules and, accordingly, the covalent bonds are no longer completely oriented. A schematic illustration of the thermal flow in a tape and the influence of the orientation on the thermal flow are shown in Fig. 10. As a result of a loss of orientation of the covalent bond in the IP direction, the orientation of the covalent bonds logically increases in the TP direction. Thus, the portion of energy transfer that can occur along the covalent bonds in the TP direction increases while the transfer that must occur via physical bonds decreases.

However, this assumption cannot be substantiated at present. Due to the high conductivity of the covalent bonds, it is possible that a significant reduction of the IP conduction will only occur when there is a higher loss of orientation, i.e. when the first physical bonds get into the conductive path.

The reduction of air enclosures has very little effect on the IP diffusivity. Since the conduction takes place along the tapes, air enclosures between and within the tapes are almost irrelevant for the diffusivity. Due to the elongated shape of the microvoids, they are not obstacles for the energy transfer through a highly oriented SRC. Consequently, the disappearance of these air enclosures does not lead to a notable shortening of the conduction path in the IP direction. Positive influences on the IP diffusivity are, therefore, marginal and possible existing effects are completely lost by the simultaneous reduction of the orientation. A schematic model developed based on microscopic analyses is shown in Fig. 11.

Based on the surface microroughness, air enclosures between the tapes can occur even if the tapes lie directly on top of each other. Then, thermal contact only exists on a small part of the surface. The actual contact points are relevant for the heat flow and are called contact spots or alpha spots in the area of contact resistances. This effect exists at very low compacting degrees and could not be clearly detected even when using samples produced at a low pressure and low temperature.

The removal of air enclosures between the tapes and the removal of microvoids obviously have a positive effect on the thermal diffusivity in the TP direction. The microvoids within

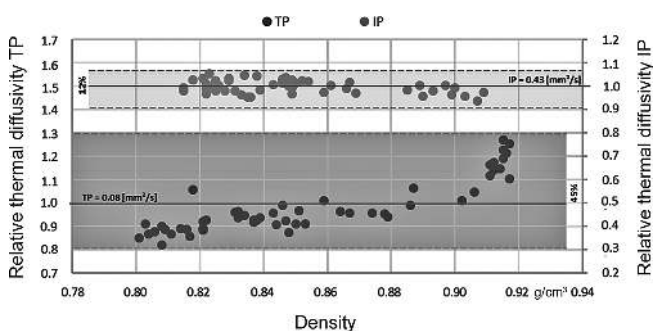


Fig. 8. Relative thermal diffusivity in the IP and TP directions in relation to the sample density

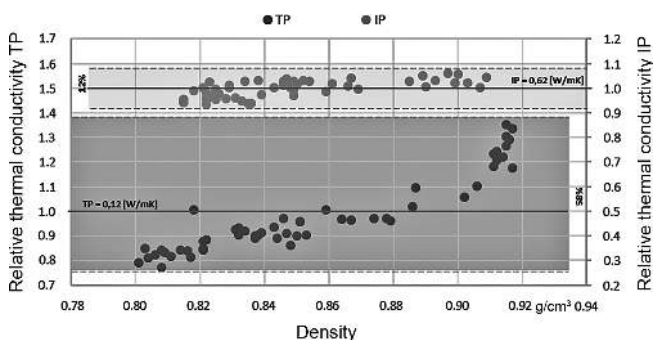


Fig. 9. Relative thermal conductivity in the IP and TP directions in relation to the sample density

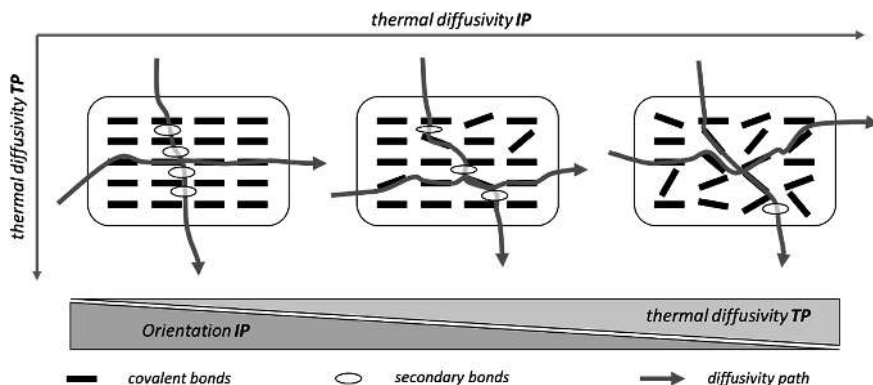


Fig. 10. Schematic illustration of the thermal flow in a tape regarding the influence of the orientation

the tapes significantly extend the TP conduction path through the sample. The heat transfer through an air inclusion is marginal. Both the double convective transition PP-air-PP and radiation effects are negligible. The latter is mainly due to the low radiation content at room temperature. As the degree of melting increases, the air enclosures are closed and, thus, become a smaller obstacle, enabling a shorter conduction path.

The same statements also apply for the effect on the thermal conductivity. There is a slight increase in the IP conductivity values, as shown in Fig. 9. The reason for this is that the reduction of the air enclosures and microvoids directly lead to a larger effective conduction area, and, as a result, to an increase in conductivity. Mathematically speaking, this aspect can also be assumed in Eq. 1 in which the density is part of the calculation.

Due to the inhomogeneity of the material, a generally valid statement cannot be made based on this. This positive aspect counteracts the effect of the lower orientation of the molecules (see Fig. 10), which is why the thermal conductivity remains almost constant regardless of the density. The TP thermal conductivity is enhanced significantly by this effect. The effective conduction area is increased by the reduction of the air enclosures (see Fig. 11), just as the conduction along the covalent bonds in the TP direction is enhanced by the melting and the associated decrease in orientation (see Fig. 10).

Since the correlations between the processing parameters and the density as well as the correlations between the density and the thermal diffusivity and conductivity are known, a parameter meant to better describe the relation between IP and

TP measurements is presented below. According to previous findings, it can be assumed that IP and TP measurements have a defined interdependence and that their ratio tends towards 1 with the increasing loss of orientation. This results in the descriptive parameter of the anisotropy, which is defined as follows.

$$\frac{\text{Thermal diffusivity IP}}{\text{Thermal diffusivity TP}} = \text{Anisotropy}(A_{\text{diff}}), \quad (2)$$

$$\frac{\text{Thermal conductivity IP}}{\text{Thermal conductivity TP}} = \text{Anisotropy}(A_{\text{cond}}). \quad (3)$$

An anisotropy of 1 represents the completely melted isotropic state. Since a sample can be measured using either the IP or TP, the assignment was made based on the density. To calculate the anisotropy, each IP or TP measured value was matched with a calculated mean value of TP or IP samples, which have the same density $\pm 1\%$. Figure 12 shows the relative density (based on 0.92 g/cm^3) in relation to the anisotropy of thermal diffusivity.

When investigating the anisotropy, it became clear that there is an almost linear relationship between the anisotropy and the relative density. The test results with the highest relative density can function as an indicator for a stronger decrease of the anisotropy above a density of 0.90 g/cm^3 . When all air enclosures have been removed from the material, the density increases up to 0.92 g/cm^3 and the relative density equals 1.

It is not clear whether the curve is hard or soft at the transition as it is shown in Fig. 12. A hard transition would be possi-

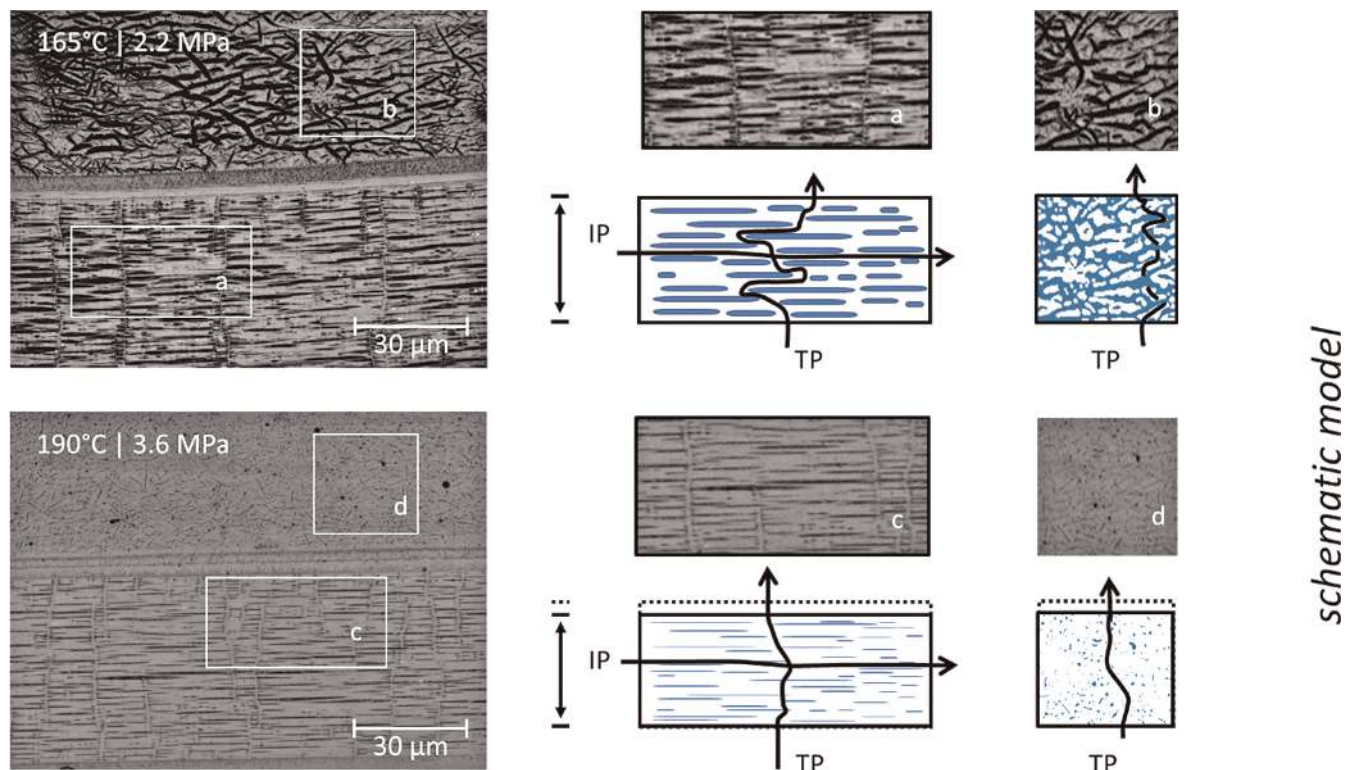


Fig. 11. Schematic illustration of thermal flow in a pressed compound

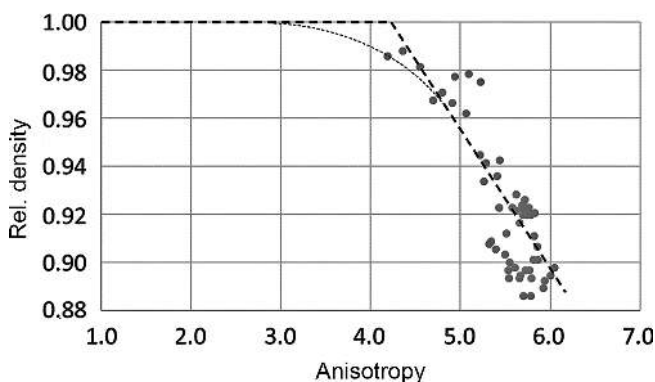


Fig. 12. Anisotropy of the thermal diffusivity in relation to the relative density (base 0.92 g/cm^3)

ble if the relative density decreases in a linear curve until it reaches 1, but also a soft transition could be present, i. e. if the included air can leave the SRC slower simultaneously with the decreasing volume. The anisotropy will further decrease after reaching a relative density of 1. Further validation of the curve by means of mapping the anisotropy in the range below 4 is not possible. Since the material displays a high degree of shrinkage at a higher degree of melting, strong deformations of the SRC in the IP direction occurred and sufficient fixation during the process was no longer given.

6 Conclusion

Previous investigations concerning the relationship between the thermal diffusivity or the conductivity focused upon elements with singular orientation (Pietralla, 1981; Kurabayashi, 2001; Hellwege et al., 1963). The aim of this study was to investigate the transferability of these interrelationships to complex fabric structures. The results show in the case of the material system used that the thermal diffusivity is a better parameter for describing the material state than the thermal conductivity. The influence of the density is less prominent here, making it possible to detect a significant correlation between the molecular orientation and the thermal conductivity.

Anisotropy can be used as the characteristic value for the description of the material condition.

Due to the high influence of the TP value on the anisotropy, only the use of this value as a parameter can be considered. This approach represents an interesting option for future material characterisations, especially since non-destructive TP measurements are easier to implement than IP measurements.

Since this study solely investigates the behaviour of the material PURE with the described microvoids, it would be advisable to evaluate other material systems in the future to evaluate the validity of the model and, if necessary, to develop a generally valid model for SRCs made from woven tapes. Due to the microvoids, which only arise within the material at higher stretching ratios during manufacturing, it can be assumed that the density cannot be used as an indicator in such a range for other material systems. A variation of the density in another

system may only be possible due to air enclosures between the tapes. Verification of the validity of such models with the method presented here remains outstanding.

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