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Commercial self-reinforced composites: a comparative study

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Abstract

Self-reinforced composites (SRCs) offer a high toughness, low cost, good recyclability, formability, and potential for hybridisation. Because matrix and fibres are of the same material, SRCs have a perfectly compatible fibre-matrix bond. Several commercial SRCs are available, but no direct comparison of these materials can be found in the literature. In this study, we therefore compared the thermal, tensile, flexural and impact of four self-reinforced polypropylene (SRPP) materials (known under the brand names Curv, PURE, Torodon and Terralys) and one self-reinforced polyethylene terephthalate material (SRPET from Comfil). PURE had the highest tensile stiffness and strength, Torodon the highest impact resistance and SRPET the highest bending strength. However, none of these materials outperformed the rest on all the tested properties, implying that the best self-reinforced composite depends on the targeted application. This paper can be used as a tool for initial screening of the commercial self-reinforced composites for a given application.

Keywords: A. Polymer (textile) fibre; B. Impact behaviour; B. Mechanical properties; B. Strength; Self-reinforced composites

1. *Introduction*

Self-reinforced composites (SRCs), also called “single polymer composites”, were invented in 1975 by Capiati and Porter [1]. They exploited the difference in melting points between extended chains of more stable crystals and conventionally crystallised melts. A significant step in the area of SRC was taken with the invention of the hot compaction process, developed at the University of Leeds by Hine, Ward and colleagues [2]. By a

suitable choice of temperature and pressure, they succeeded in melting a thin skin on the surface of highly oriented thermoplastic fibres. This skin then recrystallised to bind the fibres together and form the matrix. This research enabled the development and production of novel composites where both the reinforcement and the matrix consist of the same polymer [2]. Later, Loos *et al.* [3] developed a new technique for producing SRCs called bi-component tape technology. In this technology, the bi-component tape consists of an oriented polymer core surrounded by a copolymer with a lower melting point. The copolymer is melted to form the matrix [4,5]. Other techniques to produce the matrix of SRCs involve film stacking, powder impregnation or solvent impregnation [6-8].

One of the advantages of SRCs is their ability to achieve excellent interfaces between fibre and matrix, as both are made from the same polymer [9-11]. Another advantage of SRCs is their easier recyclability since the whole material is made of a single polymer family [12-14]. They have found widespread use in suitcase and sports applications [2].

Over the years, it became evident that polypropylene (PP) was the most suitable polymer for commercial exploitation. The first commercially available SRPP was Curv. This material is made from woven single-component oriented PP tapes [2]. Examples of commercial SRCs with bi-component PP tapes are PURE and Torodon [4]. Other polymers have also been used to produce SRCs. Polyethylene (PE) [6,7,15], poly(methyl methacrylate) (PMMA) [16], poly(lactic acid) (PLA) [12,17,18] and polyamide 6 (PA6) [19] have been used in research to produce SRCs. Poly(ethylene terephthalate) (PET) [20-22] has also been used to produce SRCs and the necessary fabrics consisting of comingled PET fibres are commercially available from Comfil in Denmark. Some natural materials have been found suitable for self-reinforcement. For example, Lu *et al.* [23] produced self-reinforced sisal composites through slight benzylation treatment, which allowed the skin layers of sisal fibres to be converted into a thermoplastic matrix.

Since their emergence, the literature devoted significant efforts to SRCs. They have been a prime candidate for hybridisation, thanks to their low cost and high toughness compared to typical structural composites based on carbon fibres [24-30]. SRPP is by far the most widely studied system. However, all these studies focus on a single type of SRC and direct comparisons between commercially available SRCs have not been found.

The properties of SRCs produced by hot compaction depend on the processing conditions used to manufacture them [2,31-34] and on the architecture of the used fabrics [35]. Direct comparisons of the properties of these materials should hence be made carefully. Some of the same materials used in this study have been studied in the literature. For example, Abraham *et al.* [36] looked into the dynamic mechanical thermal properties of autoclaved PURE SRC. They found that the tapes from this material do not show a significant glass transition due to the high molecular orientation of the tapes. This results in a tough composite under the glass transition temperature and the temperature range in which this material can be used is broader than for isotropic PP. Banik *et al.* [37] studied the flexural creep of autoclaved PURE composites. Additionally, they performed static flexural tests on cross-ply composites and reported flexural modulus and strength values of 2-4 GPa and 80-100 MPa, respectively.

Romhány *et al.* [10] found that the presence of voids in a Curv plate is reflected in the tensile load-displacement curves as small load drops (referred to as “pop-in”). Swolfs *et al.* [38] investigated the impact behaviour of Curv at and below room temperature. They found that the matrix of this material strongly changed below room temperature, leading to a reduced damaged area under impact.

Schneider *et al.* [20] studied the compression and tensile properties of SRPET. They observed that in tension, unreinforced PET is brittle, with one tenth of the failure strain of the composite SRPET. They also observed a strain-hardening effect at higher strains.

Andrzejewski *et al.* [34] studied the influence of the processing conditions on the mechanical properties of the same type of SRPET. They found that the temperature processing window for this material is quite wide. Furthermore, the relaxation of the oriented polymer under higher temperatures did not have a major effect on the mechanical properties. Instead, the impregnation of the fibres was a more important factor when processing SRPET.

This study aims to provide a comparison of five commercially available SRCs. Their thermal, tensile, flexural and penetration impact properties will be analysed and benchmarked.

2. Materials and methods

2.1. Materials

All SRCs were manufactured in house based on fabrics. Five balanced, unconsolidated fabrics were therefore sourced: four to manufacture SRPP and one to manufacture SRPET. The fabrics for SRPP were: Curv from Propex Fabrics GmbH (Germany), PURE from DIT B.V. (the Netherlands), Torodon (formerly called Armordon) from Don & Low (UK), and Terralys LF 15 from Beaulieu Technical Textiles (Belgium). The fabric for SRPET was sourced from Comfil (Denmark). Table 1 summarises the fabric characteristics.

The fabrics for Curv and Terralys consisted of homopolymer PP tapes and were suitable for the hot compaction technology. The fabrics for PURE and Torodon were composed of bi-component tapes. The fabric for SRPET was composed of commingled yarns, consisting of higher melting temperature oriented PET fibres and lower melting temperature co-PET fibres. The co-PET fibres will melt to form the matrix.

The fabric for Terralys (abbreviated to “Ter.” in some plots) is composed of tapes with seemingly two different widths. However, observation of the tape cross-section under optical microscope revealed that the narrow tapes are folded along their length, which is not

noticeable with the naked eye. Furthermore, measurement of the tape linear densities revealed approximately the same values for both tapes. Therefore, henceforth the folded tapes will be called “narrow”, while the unfolded tapes will be called “wide”. The tapes do not seem to be distributed with any regularity, but the number of wide and narrow tapes is approximately the same in both directions. The tapes from the fabrics for Curv also had folded tapes in both directions, but their properties were found to be the same and hence will not be separated out here.

Table 1. Summary of the fabric characteristics.

Name	Curv	Terralys	PURE	Torodon	SRPET
Material	PP	PP	PP/co-PP	PP/co-PP	PET/co-PET
Architecture	twill 2/2	twill 2/2	twill 2/2	plain	twill 2/2
Reinforcement type	tapes	tapes	tapes	tapes	fibres
Areal density [g/m^2]	140	133	103	108	764
Tape/yarn linear density [tex]	110	112	110	130	459
Tape/yarn spacing [yarns/cm]	6.4±0.2	5.9 ± 0.3	4.5 ± 0.1	4.0 ± 0.1	8.0 ± 0.2

2.2. Production

All plates were produced through hot pressing on a Fontijne Grotnes TP400 press. The fabric layers were placed between two aluminium plates of 320x320 mm^2 and placed in a press that was preheated at the desired processing temperature. The plates were then pressed with a certain pressure, left at the processing temperature for a certain dwell time, and afterwards left to cool down under pressure. Table 2 summarises the specific processing conditions used, the number of layers and resulting plate thickness. The processing properties for Curv were chosen based on previous work by Swolfs *et al.* [32] and the processing conditions for SRPET are in the range of the conditions used by Chen *et al.* [39] and Wu *et al.* [40]. The conditions for PURE were chosen in accordance with the manufacturer’s recommendations. From differential scanning calorimetry (DSC) analysis, it was found that the thermal properties of Torodon tapes are similar to PURE, therefore the same processing

conditions were chosen. No recommendations were found for the processing of the Terralys fabric, therefore the Curv conditions were also used for Terralys, given its similarity to Curv.

Table 2. Summary of processing conditions and plate characteristics.

Material	Curv	PURE	Torodon	SRPET	Terralys
# of layers	12	7	4	4	8
Temperature [°C]	188	150	150	220	188
Pressure [bar]	39	30	30	15	39
Dwell time [min]	5	5	5	1	5
Time to cool down to 40°C [min]	±5	±4	±4	±6	±5
Panel thickness [mm]	1.24±0.05	0.89±0.02	0.49±0.01	2.09±0.03	1.09±0.04

2.3. Differential scanning calorimetry (DSC)

DSC was performed on a DSC 3+ machine from Mettler Toledo. The tests were performed from -50°C to 250°C for all materials except for SRPET, which was found to melt close to 250°C. Therefore, the tests for SRPET were performed from -50°C to 300°C. An aluminium pan of 40 µl was used. All tests were performed at a rate of 10°C/min in a nitrogen environment. At least three samples were tested for each material.

The tests were performed on unconstrained tapes. It is known that oriented polymers have a higher melting temperature than their isotropic counterpart, and this feature is exploited to produce SRCs [41]. When performing DSC tests on unconstrained oriented tapes, due to relaxation effects upon reaching the glass transition (T_g), the obtained curves do not entirely reflect the thermal behaviour that the tapes exhibit during composite production, when they are constrained by the pressure applied during hot-compaction [3,41,42]. However, DSC tests on constrained tapes and fibres are difficult to perform in a consistent manner and were therefore not performed in this study.

2.4. Dynamic mechanical thermal analysis (DMTA)

DMTA tests were performed on Q800 device from TA Instruments. Single cantilever tests were performed in a nitrogen environment on specimens with a nominal width of 10 mm

and at a length of 17.5 mm. The tests were performed at a frequency of 1 Hz and a strain of 0.05%. After equilibrating at -50°C for 5 min, the temperature was increased at a heating rate of 2°C/min.

2.5. Single fibre/tape tests

Single tape tests were performed on tapes extracted from Curv, PURE, Torodon and Terralys fabrics, and single fibre tests were performed on fibres extracted from yarns of the SRPET fabric. These tests were performed on an Instron 5943 with a 1 kN load cell according to the ASTM C1557 standard, except for the strain measurement. Depuydt *et al.* [43] have shown that digital image correlation (DIC) improves the results achieved with this test compared to using the compliance-corrected machine displacement. Dots were therefore placed on the tape surface using a permanent marker. These dots were used to measure the average strain on the tape surface with DIC. Since marking fibres was not straightforward, instead two small epoxy resin droplets were placed on the PET fibres to track their separation during testing. This approach yielded more noise in the strain data than for the tapes due to small vibrations of the resin droplets used to track the strain. The tests were performed with a gauge length of 100 mm at a rate of 5 mm/min. At least six tests were performed on each reinforcement.

2.6. Composite tensile tests

Tensile tests were performed on the SRCs according to the ASTM D3039 standard. The tests were performed on a Zwick Z100 with a 250 kN load cell. The strain was recorded using a video extensometer, which tracked the separation of two markers on the specimens observed with a video camera. The specimens had dimensions of 250x25 mm^2 and a gauge length of 150 mm. The test rate was 2 mm/min. Four samples were tested for each material.

2.7. 3-point bending tests

3-point bending (3PB) tests followed the ASTM D7264 standard. The tests were performed on an Instron 5567 and Instron 4467, both equipped with a 5 kN load cell. The specimens had a dimension of 75x15 mm^2 and a span-to-thickness ratio of 32 was used. The tests were performed at a displacement rate of 3.3 mm/min. Four tests were performed on each material. Since none of the materials failed in a clear way, the flexural failure strain was defined as the strain at the maximum bending stress.

2.8. Penetration impact tests

For the penetration impact tests, a Fractovis CEAST 6789 Instrumented falling weight machine was used. The ISO 6603-2 standard was followed, and the tests were performed at room temperature. The specimens were clamped with a force of approximately 7900 N, leaving a circular opening with a diameter of 40 mm. A striker with a 20 mm diameter was used to impact the specimens. All specimens were impacted with an energy of 256 J by a striker weighing 26.17 kg and falling from a 1 m height. Although the ISO 6603-2 standard prescribes the half-peak method to compare the impact resistance, the full energy method gives a more complete comparison of this property, and therefore this method was chosen in this study. At least six samples were used for each material.

The shape parameter, which is sometimes called ductility index [24], was used to quantify the ratio of the propagation energy to the initiation energy. The initiation energy is the energy up to the maximum force, while the propagation energy is the energy from the maximum force to the point when the force becomes zero.

3. Results

3.1. Thermal behaviour

DSC measurements were performed on tapes and fibres extracted from the fabrics prior to processing. Figure 1 plots representative DSC curves performed on the unconstrained

fibres/tapes extracted from each of the materials. SRPET shows an enthalpy relaxation peak at 75°C and a melting peak at 251°C. These results are in agreement with results reported by Andrzejewski *et al.* [34] for the same PET fibres. All the PP tapes, except Curv, display complex melting peaks, which point to a wider range of molecular orientation in the tapes or more than one polymer type.

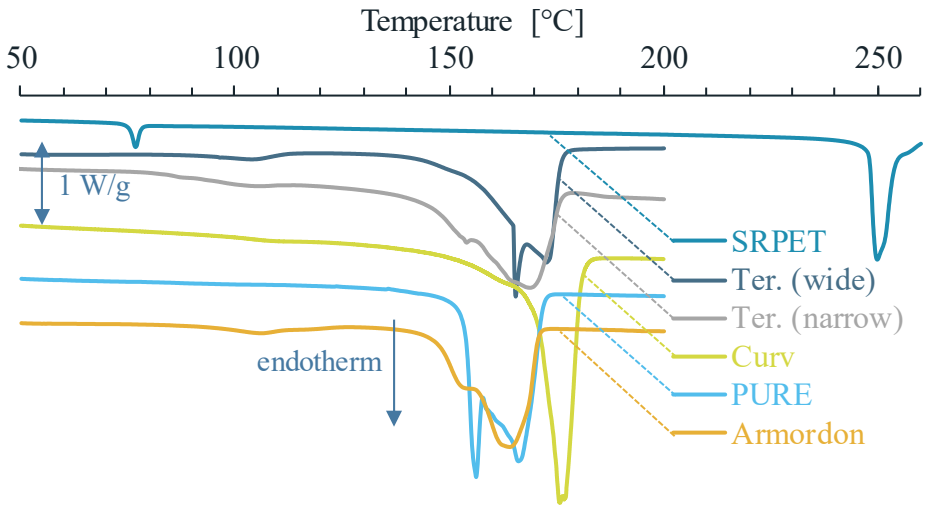

Figure 1. Representative DSC curves for each of the tapes and fibre from the fabrics.

Figure 2 displays the temperatures corresponding to the largest endotherm for each material, as well as the melting enthalpy, given by the area underneath the endotherm. All PP materials display similar peak melting temperatures, with Curv being slightly higher than the others. The melting enthalpy does not seem to follow any particular trend. SRPET clearly has the lowest melting enthalpy, indicating a lower degree of crystallinity than PP-based SRCs.

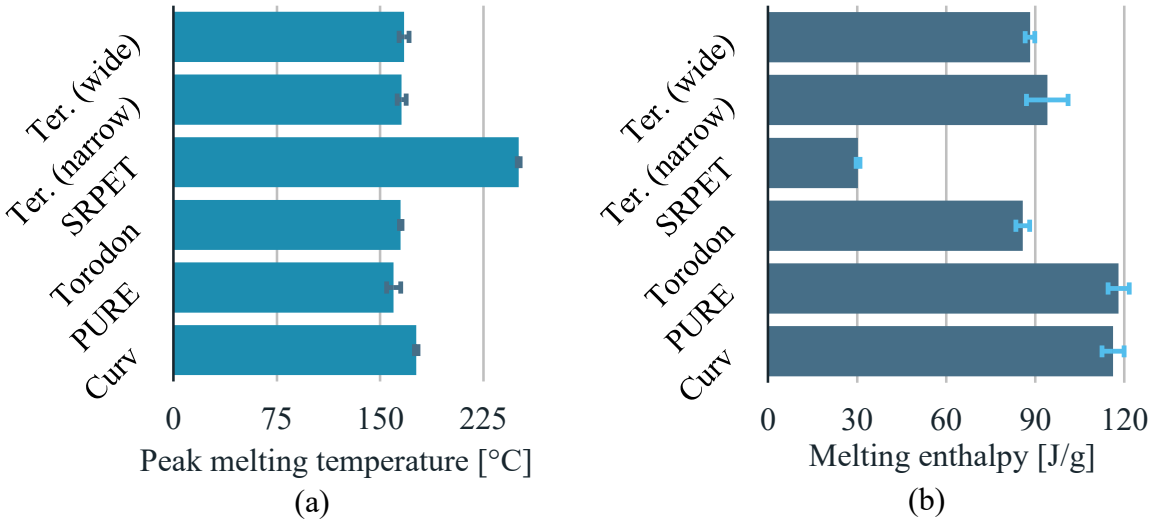

Figure 2. (a) Peak melting temperatures for each material, and (b) corresponding melting enthalpies.

Both types of Terralys tapes were analysed separately using DSC. Figure 1 reveals that although the curves have different shapes, the melting enthalpy and peak melting temperature (see Figure 2) are not statistically different, with a p-value of 0.12 and 0.43, respectively. This p-value is the outcome of a double-sided t-test. The difference in recrystallisation enthalpy of both tapes was found to be statistically insignificant (p-value: 0.17), which suggests that both tapes are made from the same grade of PP and have the same degree of molecular orientation.

Figure 3 depicts the results of the DMTA tests in terms of storage modulus and tangent of the phase angle δ. Curv and Terralys behave very similar, with a gradual reduction in storage modulus and a tan(δ) peak at 10-15°C. This peak is related to the glass transition temperature of the PP matrix. While PURE and Torodon have a similar gradual decrease, their tan(δ) peak appears around 90-100°C. The lack of the typical PP-Tg can likely be attributed to the low matrix fraction in these SRPPs compared to Curv and Terralys. SRPET reveals significantly different behaviour. Below 60°C, the matrix of the SRPET is in the glass state, and hence reasonably stiff. When the matrix goes through its glass transition around 60°C, the storage modulus drops precipitously due to the high fraction of amorphous phase. SRPET is therefore

the only of the five studied SRCs with a clear maximum operating temperature based on the DMTA results.

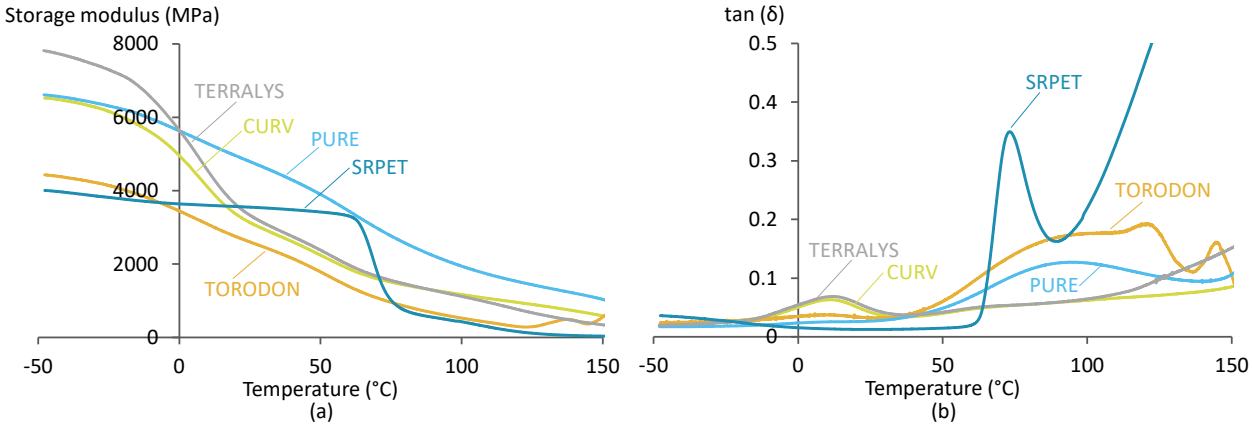

Figure 3. Representative DMTA results: (a) storage modulus and (b) tangent of the phase angle δ.

3.2. Single fibre/tape properties

Figure 4 plots representative stress-strain diagrams of the single fibre/tape tests. All tapes and fibres display non-linear behaviour: all tapes show a stiffer initial stage, whereas the SRPET fibres show at least three distinct slopes, with a stiffening region before reaching the maximum stress. This stiffening is likely attributed to increased alignment of the amorphous regions with increasing strain. The comparison of these results is, however, not straightforward for several reasons. Firstly, for the Terralys and Curv fabrics, part of the oriented polymer will be melted to become the matrix in the SRCs, whereas the copolymer on the surface of the PURE and Torodon tapes will form the matrix. The test, however, takes into account the entire cross-sectional area of the fibres/tapes used to calculate the stress. Secondly, the oriented molecules will partially relax during processing and hence have different properties in the SRCs [38]. The results from the single fibre/tape tests therefore provide only indirect indications for the tensile properties of the corresponding SRCs.

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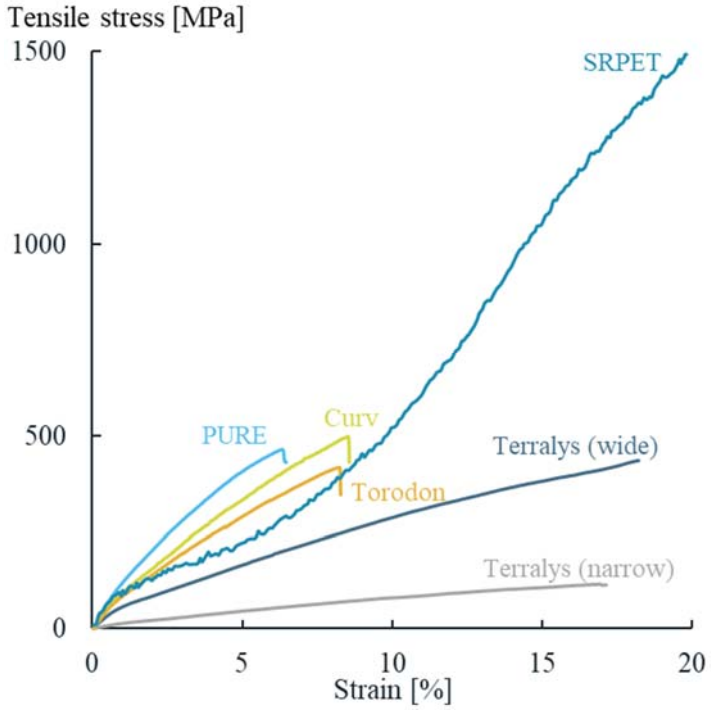

Figure 4. Representative stress-strain of single fibre/tape tests.

The fibre/tape stiffness was similar for all materials except for Terralys. The stiffness of the two different widths of Terralys tapes differ significantly (p-value: 10^{-8}) and are lower than for the other materials. All the tapes fibrillated when tensile failure occurred, whereas the PET fibres did not fibrillate. The PET fibres did display the highest strength. All tapes show a similar tensile strength apart from the narrow Terralys tapes. The failure strains for all fibres/tapes (see Figure 4) are significantly higher than for typical carbon and glass fibres, which have failure strains in the range of 1-2% and 2-4%, respectively. Terralys tapes and PET fibres had a significantly higher failure strain than the other reinforcements.

3.3. Tensile and bending properties

Figure 5 plots representative stress-strain curves of the tensile tests performed on the SRCs. The results on SRCs do not directly correlate with the tensile tests on the fibres/tapes. However, the shape of the tensile diagrams for the reinforcements (see Figure 4) is reflected in the tensile tests of the SRCs. The distinctive shape of the PET fibre behaviour remains present in SRPET (see Figure 5).

The tensile properties for SRPET are in line with the literature [20,39,40]. The small differences can be attributed to the different processing conditions used for compaction of the plates, as these conditions can have a significant influence on the properties of the composites [32]. The results for SRPET reveal striking differences with the behaviour of the PET fibres (see Figure 4), especially in terms of tensile strength. It remains unclear why this difference is larger for SRPET than for the PP-based SRCs, but it could be for a range of reasons, such as crimp in the woven fabric, hydrolytic degradation of the PET [21] or a large fraction of co-PET melting to form the matrix.

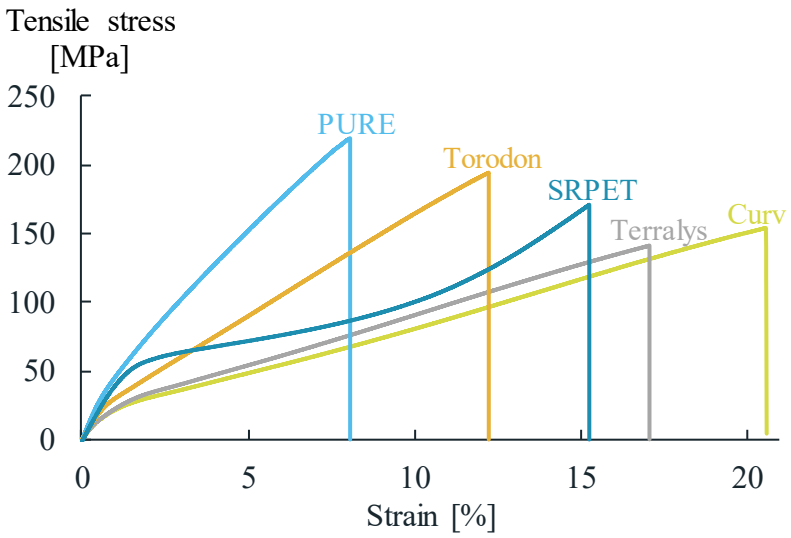

Figure 5. Representative tensile stress-strain curves for each of the tested SRCs.

The shape of the bending stress-strain plots does not reflect the shape of the plots from the fibre/tape tensile tests (see Figure 6). None of the materials reached flexural failure, so the tests were stopped after the maximum bending stress was reached.

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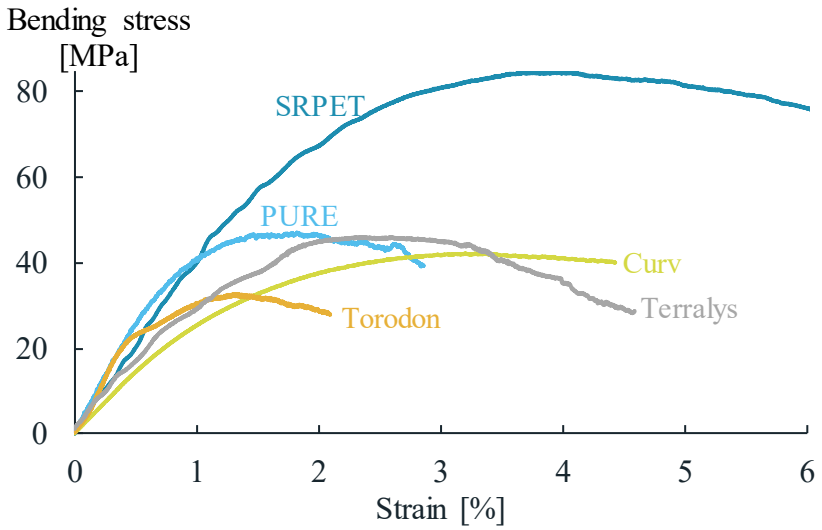

Figure 6. Representative bending stress-strain curves of different SRCs.

Figure 7 compares the tensile and bending stiffness for each SRC to the single fibre/tape results. The bending and tensile stiffness for all materials are similar, with Curv and Terralys being on the lower end and PURE and Torodon on the higher end. This observation likely indicates a higher draw ratio or more specifically a higher degree of molecular orientation in PURE and Torodon.

Torodon and Terralys display a slightly higher bending than tensile stiffness. The difference for Terralys is, however, not statistically significant (p-value: 0.38). The difference for Torodon is significant and unexpected (p-value: 0.007). One potential explanation could be an unequal distribution of the tapes over the thickness.

It is also clear that a one-to-one relationship between stiffness of the SRC and that of the corresponding single fibre/tape does not exist. While the Curv tapes have a stiffness similar to Torodon and PURE tapes, the Curv SRC stiffness ends up significantly lower. This finding is attributed to the relatively high processing temperature of Curv, causing more of the oriented tape to melt and relax than for Torodon and PURE. A similar conclusion applies to the PET fibres: although they have the highest average stiffness, the SRPET does not have the highest stiffness of the SRCs.

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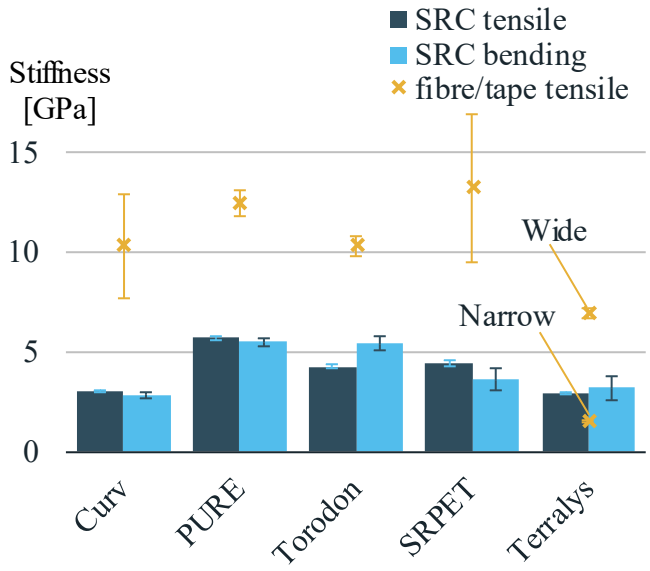

Figure 7. Stiffness results for tensile and bending tests on SRCs and for single fibre/tape tensile tests.

Figure 8 compares the tensile and bending strength for each SRC to the single fibre/tape results. The bending strengths are significantly lower than the tensile properties. This is expected, as the bending strength is significantly influenced by the compressive strength, which tends to be lower than the tensile strength for polymer fibres and their composites [20,44]. SRPET shows a significantly higher bending/tensile strength ratio compared to other SRCs. Schneider *et al.* [20] has found that the compressive strength of SRPET (94.7±0.7 MPa) is about 1.8 times lower than its tensile strength (168±10 MPa). For Curv, Mesquita *et al.* [44] found that the compressive strength of Curv can be up to 4 times lower than its tensile strength. The other SRPP materials are expected to be in line with the results for Curv, showing that this is an inherent drawback of SRPP compared to SRPET. The higher bending strength of SRPET may be attributed to two factors. Firstly, the matrix of the SRPET likely has a higher matrix stiffness, which plays a key role in compressive strength. Secondly, the SRPET fibres may be better in compression due to their lower degree crystallinity, as deduced from the smaller melting enthalpy in Figure 2b. Based on the results here and the literature, we cannot deduce which of these two factors dominates.

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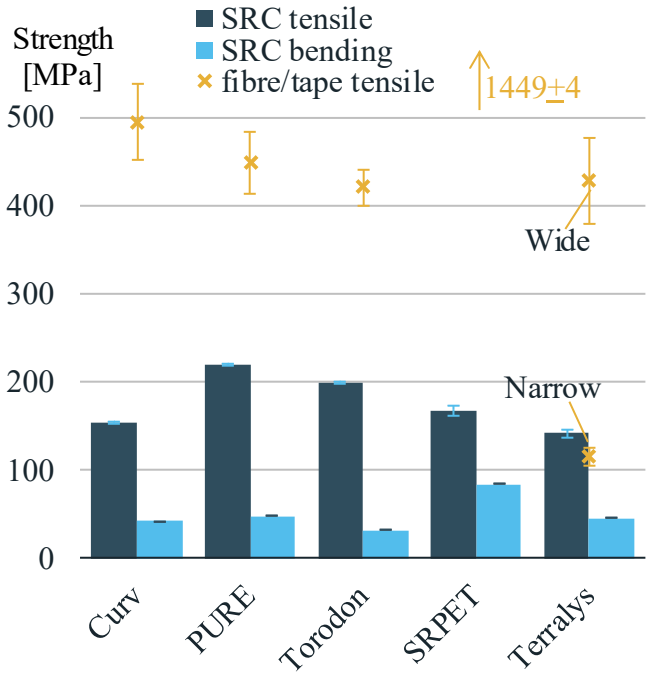

Figure 8. Strength results for tensile and bending tests on SRCs and for single fibre/tape tensile tests. The single PET fibre results are not directly shown, as they are too far out of the scale for the rest of the results.

Figure 9 compares the tensile and bending failure strains for each SRC to the single fibre/tape failure strain. The bending failure strains are difficult to compare to, as the specimens did not show bending failure.

The expectation would be that the SRC tensile failure strain is higher than the single fibre/tape failure strain. The molecular relaxation occurring during processing indeed leads to this effect for Curv, PURE and Torodon, but not for SRPET and Terralys. The observed differences are, however, small. The largest difference is observed for Curv, as the SRC has a failure strain that is twice as ductile as its tapes. This difference is again related to the high processing temperature, causing more molecular relaxation than for the other SRCs. This argument should also apply to the Terralys tapes, but it remains unclear why this was not the case. For SRPET, it could be that hydrolytic degradation during processing has embrittled the material [21], which would counteract the effect of molecular orientation loss.

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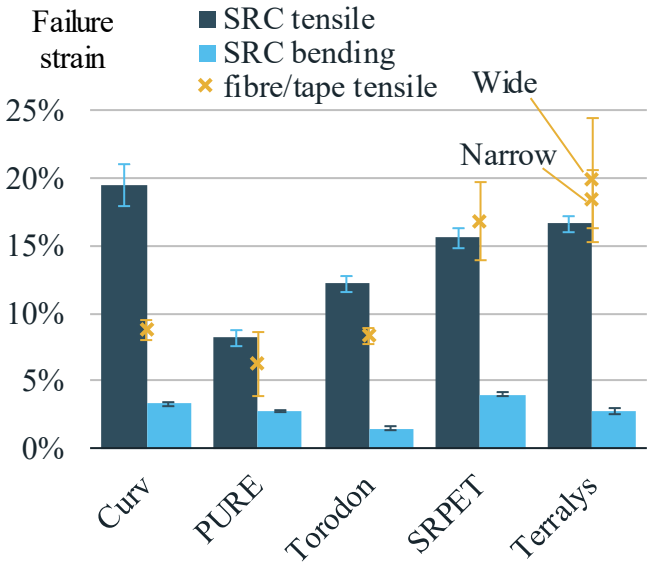

Figure 9. Failure strain results for tensile and bending tests on SRCs and for single fibre/tape tensile tests.

3.4. Impact properties

Force-displacement plots of the impact tests are presented in Figure 10. Because the specimens have a significant range of thickness, the force was normalised by the specimen thickness to allow a meaningful comparison. While such normalisation is not correct for all material types, it was found to work well for SRCs [45].

All materials, except SRPET, reveal the same general impact force-displacement plot shape, with a sharp decrease of the force after the maximum is reached, and a relatively low propagation energy. This is not the case for SRPET: it shows almost the same area under the curve before and after the peak force. This is captured clearly in Figure 11: SRPET has a shape parameter close to 1, which means that crack initiation takes almost the same energy as crack propagation. SRPET is also the only material composed of fibres rather than tapes. Nevertheless, SRPET displays a fracture surface that is similar to the other four materials (see Figure 12). All the other materials display a lower shape parameter, which is visible in Figure 10 as a sudden drop after the peak load is reached.

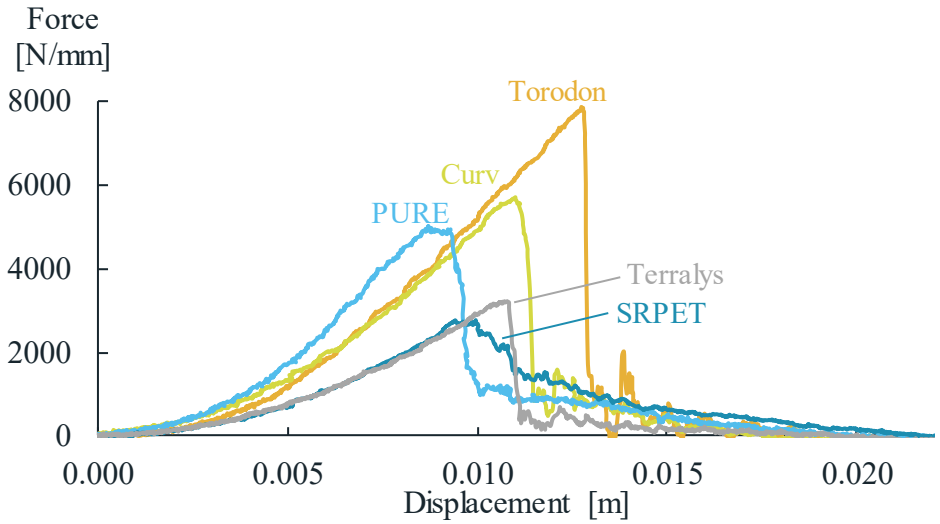

Figure 10. Representative force-displacement plots from impact testing of the SRCs. Curves were normalised by the specimen thickness.

Torodon absorbs the most amount of energy under penetration impact, and also has the most extensive visible damage (see Figure 12). However, Torodon is also the material with the lowest shape parameter (almost 0), which means that after the crack is initiated, it takes very little additional energy to fracture the material. This finding further confirms that ductility index would be a misleading terminology for the shape parameter.

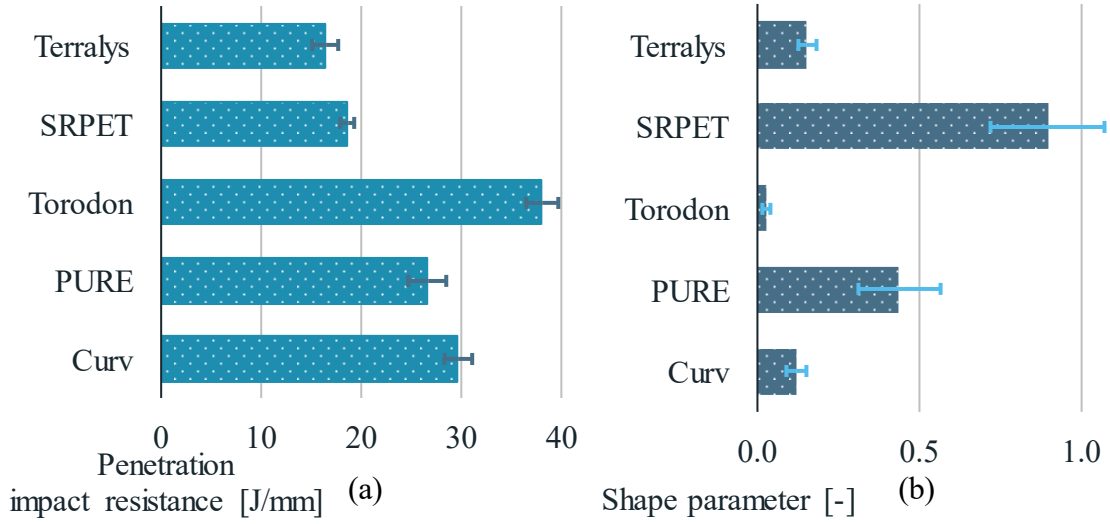

Figure 11. (a) Penetration impact resistance (from the full energy method) and (b) shape parameter.

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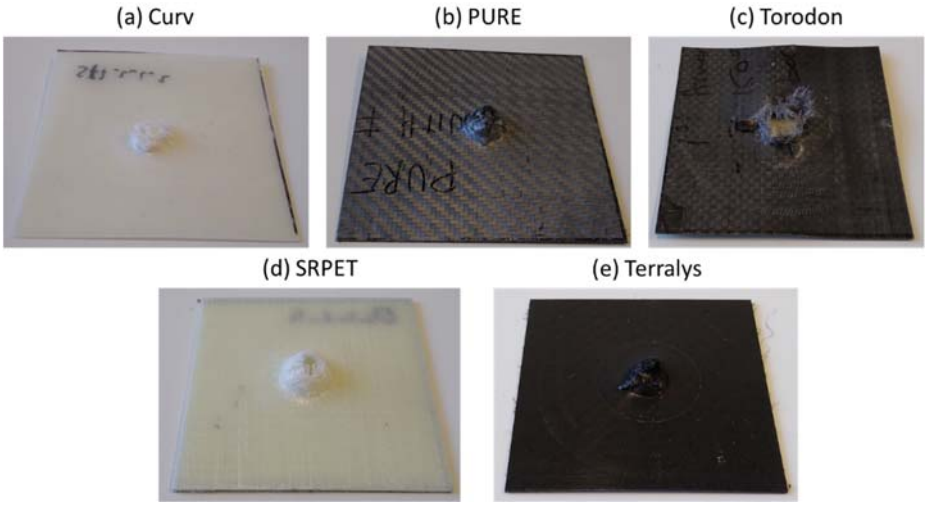

Figure 12. Photographs of representative specimens after penetration impact: (a) Curv, (b) PURE, (c) Torodon, (d) SRPET and (e) Terralys.

4. Discussion and conclusions

Five commercially available SRCs were tested in tension, 3-point bending and penetration impact. Furthermore, the thermal properties of the polymers were characterised, and the reinforcements tested in tension. Four SRPPs were tested and compared: Curv, PURE, Torodon and Terralys; as well as an SRPET material. Figure 13 summarises and compares the tensile, bending and penetration impact properties of the SRC. Every SRC has its benefits and drawbacks and Figure 13 can be used as a tool to make an initial selection depending on the application requirements. It should however be noted that other features should also be taken into account, such as formability [46], delamination resistance and temperature resistance. SRCs with a lower stiffness and strength are likely to have a better delamination resistance [47], as they are composed of lower orientation fibres/tapes. The temperature resistance is often quoted as a benefit of SRPET over SRPP. Although the use of SRPET is clearly limited to temperatures below 60°C(see Figure 3), the maximum working temperature for SRPP is less clearly defined.

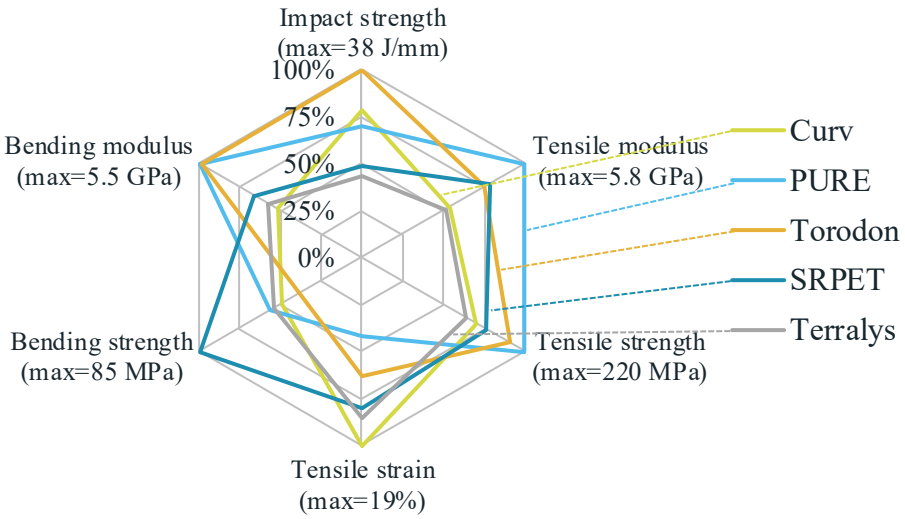

Figure 13. Comparison of the tensile, bending and impact properties of the tested SRCs (normalised by maximum values among these SRCs).

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