

High Temperature Resistant Insulated Hybrid Yarns for Carbon Fiber Reinforced Thermoplastic Composites

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ABSTRACT: With the increased use of carbon fiber reinforced composites (CFRC), the demand for the integration of insulated conductive wire/yarns in CFRC is increasing for additional function integrations such as sensoric, actoric, signal transfer, heating, etc. Between thermoset and thermoplastic matrix composites, the integration of insulated conductive materials is comparatively difficult due to the requirements of higher temperature and pressure during the consolidation of thermoplastic composites. Therefore, the need for insulating material able to withstand higher temperature for the use in thermoplastic CFRC is also high. Using DREF friction spinning technique, it is possible to manufacture yarns with a core-sheath structure in which, as the core conductive wire/yarns and as the sheath different fiber formed materials can be used for the insulation of the core. In this study, the aspects of using different short/staple fibers such as polyester, Glass and Kynol as the sheath and the usable temperature range are revealed. Furthermore, the insulation property of such fibers after the application of different temperatures has been reported. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 130: 1179–1184, 2013

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INTRODUCTION

Because of their unique combination of low-weight, high strength and stiffness properties, Carbon Fibre Reinforced Composites (CFRC) are increasingly being used as load-bearing structures for lightweight components in land, sea, air, and space vehicles as well as for many industrial, consumer and sporting goods. The demand of endless carbon fiber reinforced composites in 2011 was 39,000 tonnes and is increasing rapidly worldwide. The growth in endless carbon fiber reinforced composites up to 2020 is expected to be more than 17% per year.¹

Textile composites are composed of textile reinforcements combined with a binding matrix (usually polymeric). Depending on the type of matrix, textile reinforced composites can be divided often into two groups such as thermoset and thermoplastic composites. Though textile-reinforced composites are preferentially manufactured based on a thermoset matrix, thermoplastic matrix-based composites have now been developed due to some distinctive advantages over thermoset composites, such as lower density, unlimited storage of preforms, semi-products delivered ready for use, thermoformability, a faster processing cycle, no solvent emissions during the processing stage, recyclability, improved shock/impact behavior, and environment friendliness.² A large range of tough thermoplastic matrix materials is also available. As a result, textile-reinforced thermoplastic

composites are attracting growing interest from both the academic community and industry.³ With an increasing demand and usage of the CFRC, the issue of additional function integration e.g. sensoric, actoric, heating, signal transfer etc. in CFRC is gaining significance.

Using different textile processes such as knitting, weaving or stitching, it is possible to integrate different yarn formed functional components such as conductive filaments, yarns, metallic wires, fiber optic sensors etc. into fiber-reinforced textile preforms destined for thermoplastic composites.^{4,5} To prevent electrical short circuits, it is necessary to completely and homogeneously encompass the conductive material with an insulation layer for the integration of different conductive yarns/wire into CFRC. However, for the manufacturing of thermoplastic composites, the required insulation properties need to be guaranteed after the consolidation process of the composites (i.e., the ability of the insulation layer to withstand the temperature and pressure required during consolidation). The temperature required for the consolidation depends on the type of thermoplastic fiber used, e.g. for Polypropylene (PP) around 220°, Polyester (PES) 275°C, and Polyetheretherketone (PEEK) 380°C.⁶

Conductive wire is typically covered or coated with an insulating material, for example, a polymeric material to provide

electrical insulation. One technique for obtaining thin coatings involves dissolving or suspending polymeric materials, especially low molecular weight polymeric materials, including for example low molecular weight polyurethanes, polyesters, polyamides, or polyesterimides in an organic solvent, such as xylene, cresylic acid, or phenols. The polymeric material is typically dissolved or suspended in the solvent at a concentration of about 25% by weight. The wire is coated by being passed through the polymeric solution. The coated wire is then passed through a furnace to flash off the solvent. This cycle is necessarily repeated several times, and generally as many as seven times, to obtain a coating having a desirable thickness. The higher concentrations of polymer permits the application of increased amounts of polymer during each pass, thereby reducing the total number of pass. However, the use of higher concentrations of polymer typically results in the application of a less uniform coating. These solvent based techniques are undesirable as well due to the fact that the organic solvents which are used, for example phenol and cresylic acids generally pose numerous health and environmental concerns. To obtain desirable heat resistance, it is often necessary to prepare insulating coatings from high molecular weight materials. However, insulating coatings prepared from high molecular weight materials generally possess high viscosities relative to coatings prepared from low molecular weight materials. Accordingly, there is increased difficulty associated with handling such coatings and they are often applied to wires in the form of thick layers leading to stiff materials with problematic processability. Desirable heat resistance has also been provided in the prior art with highly crosslinked polymeric materials. Such highly crosslinked materials tend to be brittle and typically crack during working of the wire.⁷ Furthermore, the application of such polymer coatings often increase the fiber-fiber and fiber/metal coefficient of friction and thus reduce the process ability of coated wire/yarn.

In order to overcome the problems associated with the polymeric coating of conductive wire, the concept of hybrid yarns that have a core and sheath structure, fulfil to a great extent, the aforementioned conditions for the insulation of conductive wire/yarns to be used as textile integrated functional component in thermoplastic CFRC. With the DREF-2000 friction spinning process, it is possible to insulate conductive wire/filaments with a sheath. Dr. Ernst Fehrer developed and commercialized the friction spinning system under the name DREF (DR Ernst Fehrer - DREF) in the 1970s.⁸ The invention promoted a renewed interest for the research works and composite industry in core yarns. The DREF-2000 friction spinning machine is one of the latest developments in friction spinning. In this system, the yarn formation takes place with the aid of frictional forces between rotating drums rotating in the same direction. A major advantage of the DREF-2000 friction-spun core yarns is the accurate positioning of the wire/filament at the centre of the yarn cross-section with a desired level of core coverage by the sheath made of short staple fibers. The manufacturing of different conductive core hybrid yarns using friction spinning techniques for different functional applications e.g. as a sensor for structural health monitoring, signal transfer and electromagnetic shielding has been reported in different articles.^{9–11}

However, the aspect of using insulated hybrid yarn for high temperature applications in composites is not yet reported.

In the following discussion, the aspects of insulation property of different fibers used as the sheath of the conductive core hybrid yarn manufactured by the friction spinning technique, which will be able to withstand high temperature and pressure required during the consolidation of thermoplastic CFRC are described. As the core of the hybrid yarn, a carbon filament yarn (CFY) is used and the sheath with three different types of short/staple fibers such as Glass fiber (GF), PES, and Kynol is investigated. The temperature range and the core-sheath ratio, which can be used to obtain sufficient insulation for different fibers have been investigated and revealed.

EXPERIMENTAL

Materials

Material for Core. *Tenax-J HTA 40* CFY of 67 tex (number of filaments 1000, filament diameter $7\ \mu\text{m}$ and electrical resistivity $1.6 \times 10^{-3}\ \text{ohm cm}^{12}$) is used as the core component of the hybrid yarn.

Materials for Sheath. Three types of staple fibers are used as the sheath of the hybrid yarns, which are detailed below:

PES. A PES sliver of 2.6 ktex (fiber length of 28 mm) is used. The melting temperature of the fiber is around 256°C .

GF. The sliver GF of 2 ktex (fiber fineness of 2.21 dtex and 150–220 mm length) supplied by Johns Manville Europe¹³ is used.

Kynol. Kynol novoloid staple fibers *KF-0251-HC* (length 51 mm, diameter $15\ \mu\text{m}$) supplied by Kynol Europe is used. The fibers are processed to a 8.9 ktex sliver using laboratory carding machine. Kynol novoloid fibers are cured phenol-aldehyde made by acid-catalyzed cross-linking of melt-spun novolak resin to form a three-dimensional fully cross-linked, amorphous network polymer structure similar to that of thermo-setting phenolic resin. Because of their basic chemical structure Kynol fibers are infusible (nonmelting) and insoluble. Kynol[®] is a trademark and the only commercial available novoloid fibers.¹⁴

Thermogravimetric Analysis (TGA) of Sheath Fibers Used

TGA is performed to investigate the thermal stability and weight losses of PES, GF, and Kynol fibers, which are used as the sheath of the hybrid yarn in air. TGA is carried out with TA Instruments Q500 within the temperature range $30\text{--}1000^\circ\text{C}$ in air with a flow rate of 50 mL/min and a heating rate of $40^\circ\text{C}/\text{min}$.

Production of Friction Spun Hybrid Yarn

The conductive hybrid yarns are manufactured on a DREF 2000 friction spinning machine (Fehrer AG, Linz/Austria). Different yarns are produced by varying the sheath material (i.e., PES, GF, and Kynol) and fineness. The spinning parameters used for the production of the friction spun hybrid yarns include 50 m/min yarn delivery speed, 4500 rpm opening roller speed, 1500 rpm spinning drum speed and $-13\ \text{mbar}$ suction air pressure. Different yarns produced using these machine parameters are detailed in Table I.

Table I. The Friction Spun Hybrid Yarns Manufactured for the Investigations

Core	Sheath		Core: Sheath volume ratio	
	Material	Fineness		
CFY 67 tex	PES	150 tex	25:75	
		200 tex	20:80	
		300 tex	15:85	
	GF	800 tex	10:90	
		Kynol	200 tex	20:80
			250 tex	16:84

Pressing Between Two Layers of Carbon Woven Structures

In order to obtain an idea about the insulation property of the sheath material, the friction spun hybrid yarns are pressed between two layers of woven fabrics (10 cm × 10 cm) made of CFY. First of all, 6 hybrid yarns (each of 15 cm) are laid up cross-wise (3 yarns horizontal and 3 yarns are arranged vertically with a side wise distance of about 2 cm between two yarns) between two layers of the woven structures. Then, they are compressed by the laboratory press machine P 300 PV (Dr. Collin GmbH) by using different temperatures of 220, 300, and 380°C. The temperature and pressure with time used are shown in Figure 1. These are the typical consolidation programs to manufacture thermoplastic composites.⁶ For this study, the manufactured friction spun hybrid yarns are pressed between two layers of pure CFY woven structure to measure the insulation property more precisely.

Test of Insulation

For the test of insulation, a Picoammeter/voltage source (Keithley 6487) is used. The insulation of the manufactured hybrid yarns are measured before and after pressing. The insulation resistance is measured using a 10 V voltage. A metal box is used to avoid electro-magnetic effect from other sources during the measurements. Before pressing, the insulation resistance between one end of the CFY core and the sheath (distance about 2 cm) of the hybrid yarns is measured [cf. Figure 2(a)]. After pressing, the insulation resistance between one end of the CFY core of the hybrid yarns pressed between two layers of the fabrics and the fabrics is measured [cf. Figure 2(b)].

Investigation of the Effect of Temperature on the Stress–Strain Behavior of GF

In order to find the influence of temperature on the stress–strain behavior of the GF used as the sheath of the hybrid yarn, the GF is treated with a temperature of 220°C for 15 min in a muffle oven (Nobetherm Controller, B170). Then the stress–strain behavior of individual GF (using Fafegraph ME, Germany) is measured following the standard of DIN EN ISO 5079 before and after treating with temperature. Before the measurement of stress–strain behavior, the individual fiber fineness is measured by determining resonance frequency following the standard of DIN EN ISO 1973 (using Vibromat ME, Textechno, Germany).

RESULTS AND DISCUSSION

Thermal Stability of the Sheath Materials

The thermal stability of the different fibers used as the sheath of the hybrid yarns, which is measured by the TGA analysis can be seen from Figure 3. Table II shows the calculated weight loss in percentage. GF fiber shows very good temperature resistance up to 1000°C. The degradation of PES starts from 400°C. However, due to lower melting temperature (256°C), PES can not be used as insulation material at temperatures of more than 250°C. Kynol shows good stability up to 350°C. After 380°C it starts to degrade to a greater extent.

Insulation Resistance of the Hybrid Yarns Before Pressing

The results of the test of insulation of hybrid yarns before pressing can be seen from Figure 4(a). It shows that the hybrid yarns made of PES with 150 tex and 200 tex sheath possess insulation resistance of $8 \times 10^{10} \Omega$ ohm. With 300 tex sheath, it increases to $2.7 \times 10^{11} \Omega$. The hybrid yarn with GF sheath also shows higher insulation resistance, which is around $3.6 \times 10^{11} \Omega$. With 200 tex and 250 tex sheath using Kynol fibers, it is possible to obtain still higher insulation resistance which is around $1.8 \times 10^{12} \Omega$. It is not possible to obtain sufficient insulation resistance with PES and Kynol fibers with sheath fineness of less than 150 and 200 tex, respectively, due to insufficient coverage of the core.

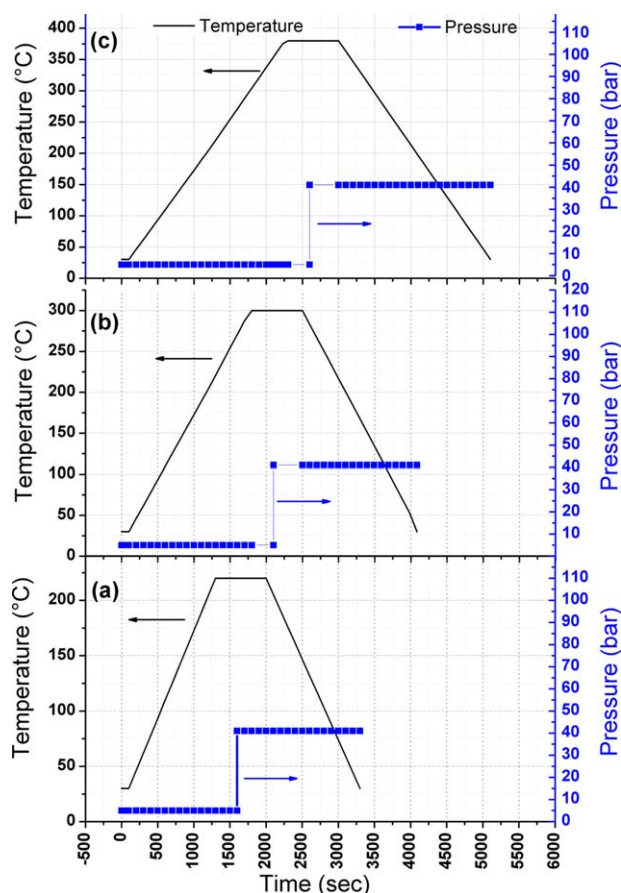


Figure 1. Temperature and pressure diagrams with time during the pressing with 220°C (a), 300°C (b), and 380°C (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

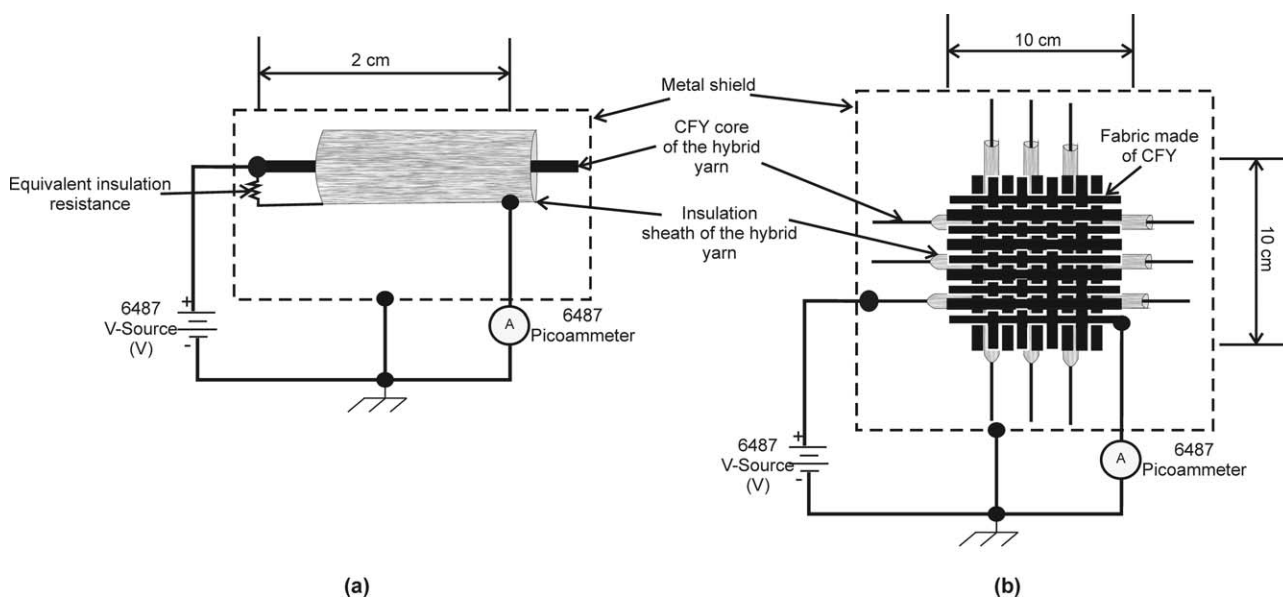


Figure 2. Test set-up for insulation test in yarn (a) and after pressing (b).

Insulation Resistance of the Hybrid Yarns After Pressing

Though the hybrid yarns, which show sufficient insulation properties before pressing, the insulation is not sufficient for many cases after pressing. The results can be seen from Figure 4(b) in case of after pressing. For hybrid yarns, which are made of PES sheath of/higher than 200 tex show very good insulation

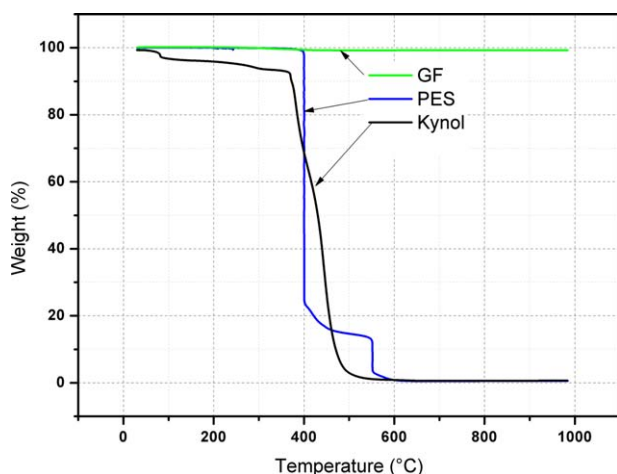


Figure 3. TGA curves of the fibers used as the sheath of the hybrid yarns. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table II. Weight Loss (%) Calculated at Different Temperatures (up to 500°C) in Air from TGA Data

Material	Weight loss (%) at								
	100°C	200°C	250°C	300°C	350°C	380°C	400°C	450°C	500°C
PES	0	0	0	0.1	0.2	0.4	7.6	83.5	85.3
GF	0	0	0	0.1	0.3	0.5	0.6	0.7	0.8
Kynol	2.2	3.7	4.8	6.2	6.8	15.0	31.2	73.4	97.0

(minimum insulation resistance of $2.5 \times 10^{11} \Omega$) when pressed at 220°C. Since the melting temperature of the PES fiber is around 256°C, the hybrid yarns with PES sheath are not tested over 220°C. It is understandable that at around 250°C, due to the melting of PES, the core will be exposed resulting in no insulation.

The hybrid yarns with Kynol with 250 tex also show similar insulation resistance in 300°C and 380°C compared to those of PES which are found for 220°C. However, with 200 tex Kynol, it is not possible to have sufficient insulation. The change of the colour of Kynol fibers of the sheath from yellow to black after pressing can be observed from Figure 5(a,b).

The yarn with GF sheath (800 tex) shows very poor insulation even at 220°C. Since the GF has higher temperature resistance compared with that of PES, it was expected that the yarns with GF would show better insulation. However, GF is brittle in nature. Therefore, it is anticipated that due to higher temperature and pressure, its brittleness increases and breaks.

Effect of Temperature on the Mechanical Properties of GF

From Figure 5(c), it can be clearly seen that the GF sheath breaks after pressing even at 220°C and exposes the CF. In order to understand the extent of the influence of temperature on the mechanical properties of GF fiber, the fibers are treated with 220°C temperature according to the time cycle as it is used

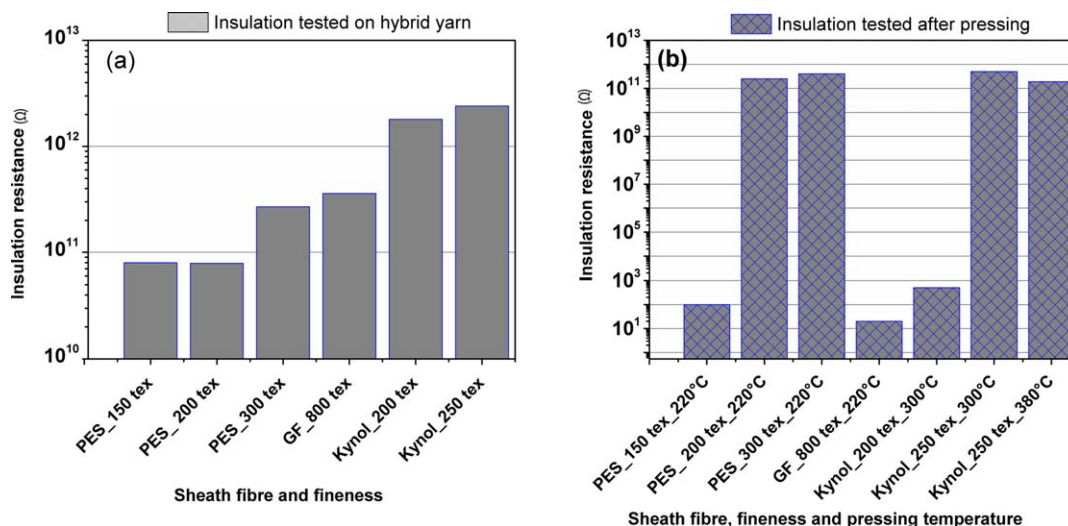


Figure 4. Insulation resistance of hybrid yarns before (a) and after pressing (b). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

during pressing. Then the stress–strain behavior of the GF before and after temperature is measured. From Figure 6, it can be seen that due to temperature treatment, the tenacity and elongation at break of GF are affected to a great extent.

CONCLUSIONS

In this study a comparative study of the use of different fibers as the sheath of the friction spun hybrid yarns to insulate the conductive CFY core is done. The results show that with PES, it is possible to get completely insulated yarns with minimum of 200 tex fineness i.e., core: sheath volume ratio of 20:80 at 220°C. Since PES melts at around 256°C, it can not be used for the insulation using more than this temperature. Though GF has higher thermal resistance compared to PES and Kynol, the insulation of GF is found very poor after pressing. Though GF

shows very good thermal resistance up to 1000°C, due to brittle nature of the GF, it breaks during pressing even at 220°C i.e. with the application of temperature and pressure. The hybrid yarns with Kynol having 250 tex sheath fineness show good potential to be used as the insulation up to 380°C. Kynol fibers are typically used as the thermal insulators and they are not high temperature materials in the usual sense of the term. However, the study shows that the Kynol fibers can be used as the sheath of conductive core to be successfully integrated into thermoplastic CFRC of temperature up to 380°C. These results also show the potential of using friction spinning technique to insulate other conductive materials such as metal wires etc. to be used in higher temperature range. In conclusion, it can be said that the results of the investigations show an easy, useful and productive way to overcome the problems to insulate the conductive materials for the integration into thermoplastic CFRC.

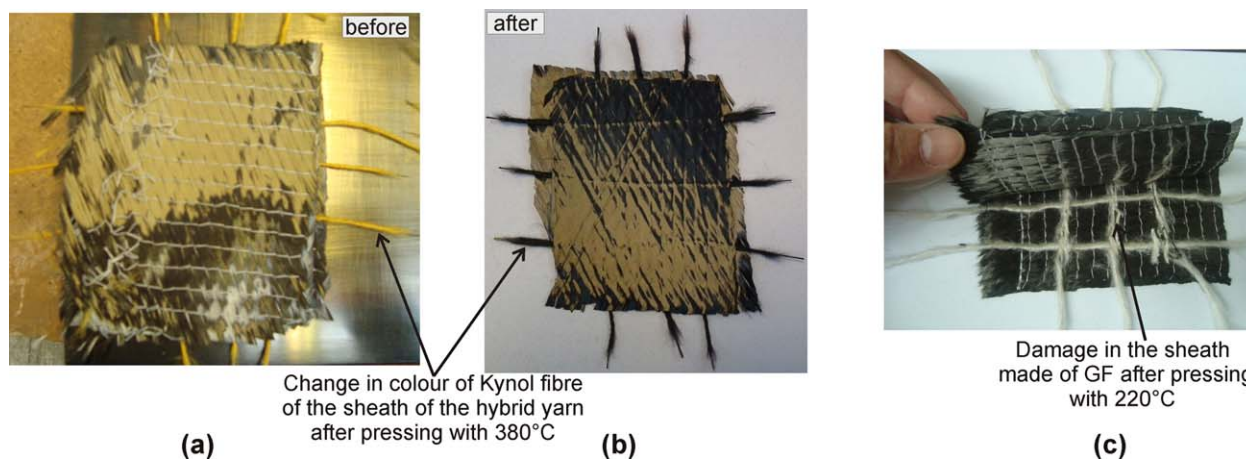


Figure 5. Test specimen before (a) and after pressing (b) with 380°C in case of Kynol fiber (shows change in colour due to temperature); (c) the breakage of GF sheath after pressing with 220°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

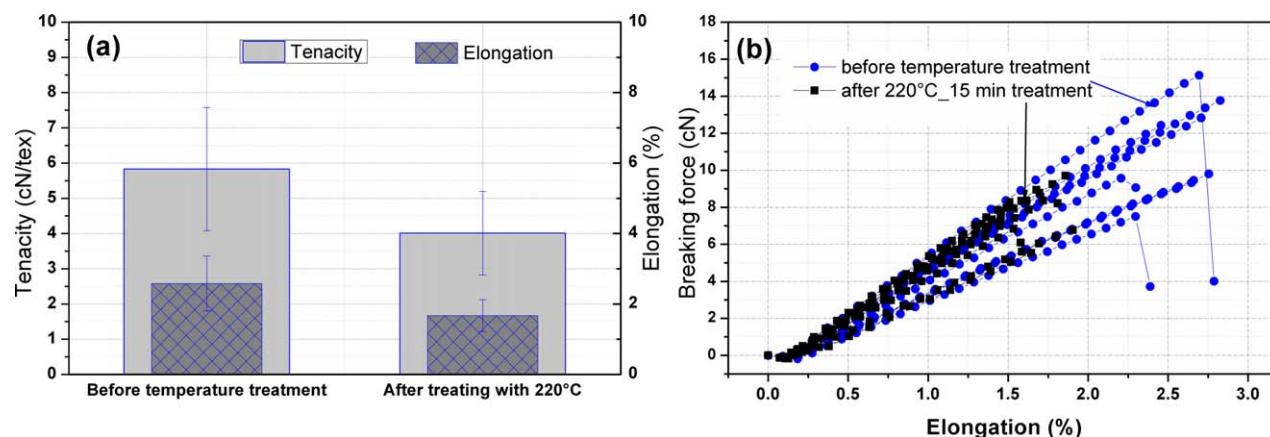


Figure 6. (a) Tenacity and elongation of GF after and before temperature treatment with 220°C for 15 min; (b) respective breaking force and elongation curves. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

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REFERENCES

- Lässig, R.; Eisenhut, M.; Mathias, A.; Schulte, R. T.; Peters, F.; Kühmann, T.; Waldmann, T.; Begemann, W. Serienproduktion von hochfesten Faserverbundbauteilen. Study, VDMA: 2012; p. 34. (German) (<http://plasticker.de/news/shownew.php?nr=17559>) (accessed 13 November 2012).
- Reyne, M. Composite Solutions, Thermoset and Thermoplastics, Ed.; JEC Publications: Paris, France, 2006; 136.
- Long, A. C. Design and Manufacture of Textile Composites; Woodhead Publishing Limited: England, 2005; 197.
- Abounaim, M.; Cherif, C. *Text. Res. J.* 2012, 82, 288.
- Mountasir, A.; Hoffmann, G.; Cherif, C.; Kunadt, A.; Fischer, W.-J. *J. Thermoplast. Compos. Mater.* 2011, 25, 729.
- Choi, B. D. PhD thesis, Technische Universität Dresden Germany, 2005. (German).
- Frihart, C. R. (Union Camp Corporation). U.S. Patent 5,786,086 (1998).
- Fehrer, E.; Fuchs, H.; König, F. U.S. Patent 4,107,909 (1978).
- Ramachandran, T.; Vigneswaran, C. *J. Ind. Text.* 2009, 39, 81.
- Hasan, M. M. B.; Diestel, O.; Cherif, C. *Text. Res. J.* 2011, 81, 1603.
- Hasan, M. M. B.; Matthes, A.; Schneider, P.; Cherif, C. *Mater. Technol.* 2011, 26, 128.
- Toho Tenax. Available at: <http://www.tohotenax.com/tenax/en/products/standard.php> (accessed 15 November 2012).
- Johns Mannville Europe. Available at: <http://www.jmeurope.com/> (accessed on 02 January 2012).
- Kynol Europe. Available at: http://www.kynol.de/pdf/kynol_flyer_en.pdf (accessed 15 November 2012).