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Knot- and Loop Tensile Tests of Ultra High-Modulus Pitch-Based Carbon Fibers

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Araştırma Makalesi / Research Article

KNOT- AND LOOP TENSILE TESTS OF ULTRA HIGH-MODULUS PITCH-BASED CARBON FIBERS

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ABSTRACT: Increasingly Fibre Reinforced Plastics (FRP) replace metal materials e.g. because of their lightweight. Besides FRP have a low thermal conductivity (TC). Thus, applications like are limited because heat cannot conduct through FRP and causes damage to electrical components.The TC of FRP can be increased one order and more using pitch-based carbon fibres. Unfortunately the processing of these fibres is still a huge challenge because of the high fibre brittleness. The Institut für Textiltechnik of RWTH Aachen University, Germany, has developed technologies to process these fibres to enable the manufacturing of 3D preforms for new ways of lightweight engineering.

Key words: Fibre Reinforced Plastics, Carbon fibre, Glass fibre, Pitch based, Thermal Conductivity

ULTRA YÜKSEK MODULLÜ ZİFT-ESASLI KARBON LİFLERİN DÜĞÜM VE HALKA ÇEKME TESTLERİ

ÖZET: Hafif olmaları gibi sebeplerle elyaf takviyeli plastikler (FRP) hızlı bir şekilde metallerin yerini almaktadır. Dahası FRP malzemelerin termal geçirgenlikleri (TC) de düşüktür. Bu sebeple, benzer uygulamaları sınırlıdır çünkü ısı FRPler ile iletilemez ve elektronik parçalara zarar verir. FRPlerin TC değerleri zift esaslı karbon elyaf kullanımı ile arttırılabilir. Ancak yüksek elyaf kırılganlıkları sebebiyle bu liflerin üretimi hala çok zordur. Almanya RWTH Aachen Teknik Tekstiller Ensititüsü, hafif mühendislik uygulamaları için 3D ön şekillerin üretiminde kullanılmak üzere bu liflerin işlenmesi için teknolojiler geliştirmiştir.

Anahtar kelimeler: Elyaf takviyeli plastikler, karbon elyaf, cam elyaf, zift esaslı, termal iletkenlik

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1. INTRODUCTION

Collaborative effort between the Institut für Textiltechnik (ITA) of RWTH Aachen University, Germany, and the Center for Composite Materials (CCM), University of Delaware, USA, has shown that the thermal conductivity of FRP can be increased one order and more using pitch-based carbon fibres [1]. Novel research from ITA and CCM demonstrates that the in-plane TC can be increased to 13 W/mK and more using around 20 % pitch-based carbon fibres of the preform in in-plane direction at 60 % fibre volume fraction (FVF). The use of overall 2 % pitch-based carbon fibres in out-of-plane direction at 60 % FVF can increase the out-of-plane TC from 0.6 to 1 W/mK using only low conductive pitch-based carbon fibres. Today the processing of pitch-based carbon fibres is still a huge challenge because of the high fibre brittleness. ITA has developed technologies to process these fibres to enable the manufacturing of 3D preforms for new applications and a new way of lightweight engineering.

Two kind of carbon fibres are of interest for industrial applications. They differ in precursor materials, polyacrylonitrile (PAN) and pitch, from which they are derived. The PAN based fibre is the most widely used carbon fibre and has been accepted widely in the market because of its high strength. In contrast, the pitch-based carbon fibre is used in special applications in which special emphasis is placed on the specific properties of the fibre. The special properties of pitch-based carbon fibres are e.g. the ultra high modulus and the high thermal conductivity. The production of pitch-based carbon fibres is conducted in several stages. It includes the production of a precursor, the precursor melt spinning, the stabilization, the carbonization and the graphitization. A surface treatment to improve the compatibility with a matrix material in case of compounds can be applied [2, 3].

The production of carbon fibres from pitch precursor is possible using a variety of intermediate stages. Therefore the production of isotropic or anisotropic pitch represents an intermediate step. Isotropic pitch is used to establish a general-purpose (GP) carbon fibre. In contrast to the anisotropic mesophase, the molecules of the isotropic pitch are randomly oriented. Thus, the generated carbon fibre is not graphitic and has poorer properties than a high-performance (HP) carbon fibre produced from mesophase pitch [4]. During the initial heating, the substance of non-volatile organic compounds melts makes isotropic pitch. If the temperature is raised about 350 °C, visually anisotropic regions appear in the isotropic matrix. These regions arise through a series of dehydrating condensation reactions among the molecules, whereby large molecules are formed, that form together a liquid-crystalline phase. This phase, called mesophase (Greek for changing phase), has a higher surface tension than the isotropic phase and is therefore of little droplets. The anisotropic mesophase droplets grow and coalesce if the temperature is increased further and hold constant over a period of time. The union of these areas continues to progress unless a continuous phase is formed [5, 6]. The formation of the mesophase was for the first time described of Brooks and Taylor [7]. With the graphitization the properties of pitch-based carbon fibres change greatly. The tensile strength and modulus of mesophase pitch-based carbon fibres increase with increasing temperature. Carbon fibres derived from isotropic pitch show a contrary behaviour. The tensile strength of isotropic pitch-based carbon fibre decreases when the process temperature is increased. PAN-based carbon fibres also show these trends [4].

2. MATERIAL AND METHOD

The thermal conductivity (TC, often referred as k) is a material's ability to conduct heat and is defined as the heat flux generated over a distance due to a steady state temperature difference. Thus, TC is the amount of heat, Q, that is transmitted in a period of time, t, over a distance, L, perpendicular to a surface of an area, A, due to a temperature difference, T, under steady state conditions (Equation 1) [8].

$$k \quad \frac{Q}{t} \quad \frac{L}{A \quad T} \qquad (1)$$

Baehr et. all defines TC as an effect of the transport of energy between neighbouring molecules based on a temperature gradient in the material. In metal materials also free electrons can transmit this energy.

The idea to manufacture FRP components, which are thermally conductive in three dimensions, offers new possibilities of light weight design – especially for the air and space industry. Unfortunately the thermal conductivity of the most common fibre materials is very low. A metal material like aluminium has a TC of 237 W/mK whereas copper – one of the highest thermally conductive materials – has one of 399 W/mK. Normally CFRPs have compared to these metals low TC values around 4-5 W/mK in in-plane and around 1 W/mK in out-of-plane direction. This is caused by both components of FRP – the matrix and the fibres. Epoxy matrix systems do have a TC of ~0.2 W/mK. Glass fibres have a low TC of ~1 W/mK whereas PAN-based carbon fibres have a TC of ~9 W/mK. However, some pitch-based carbon fibres have up to 1000 W/mK a higher axial conductivity than copper.

For this research project several pitch-based carbon fibres from Nippon Graphite Fiber Corporation, Tokyo, Japan, and Mitsubishi Plastics Incorporation, Tokyo, Japan, have been selected (Table 1).

Table 1. Selection of mesophase pitch-based carbon fibres

Fibre producer	Type of fibre	Tensile Strength [MPa]	Tensile Modulus [GPa]	Elongation [%]	Density [g/cm³]	Fila-ments per tow	Yield [g/1000m]	Thermal conductivity [W/mK]
Nippon Graphite Fiber	YSH-50A-10S	4020	519	0.7	2.1	1000	76	120
	YSH-50A-60S	3690	520	0.7	2.1	6000	532	120
	XN-90-60S	3270	886	0.4	2.19	6000	884	500
	XN-80-A2S	3300	788	0.5	2.18	12000	1797	320
Mitsubishi Plastics	K13D2U	3790	935	0.4	2.2	2000	367	851
	K63A12	3020	796	0.3	2.16	12000	1975	220

The mesophase-pitch based carbon fibres are brittle, having an ultra high tensile modulus and a very low elongation capability. The material brittleness makes it hard to process on textile machinery. However the realization of thermally conductive FRP components requires fibres that can be 3D woven, braided, stitched or tufted to realize 3D preforms. Stitching and tufting are well established processes in the textile industry. Stitching is used to fix layers together whereas tufting is used in carped production to insert the thread on a primary base. Both processes insert fibres perpendicular to the surface into a base and are useful to reinforce 2D preforms in out-of-plane direction. In case of thermally conductive FRP's this out-of-plane reinforcement can conduct heat away from the surface of the component. Finally new ways of fibre modification are required to stabilize the pitch-based carbon fibres for these processes.

First handling tests with pitch-based carbon fibres show that low bending angles must be avoided because this causes a break of all filaments (Figure 1). A bigger loop does not cause filament breakage in the first place (Figure1- 1). If the loop becomes smaller (Figure1- 2) the first filaments start to break. The knot (Figure 1-3) is already broken. Thus, only a small amount of force is required to pull the filaments apart (Figure 1-4).

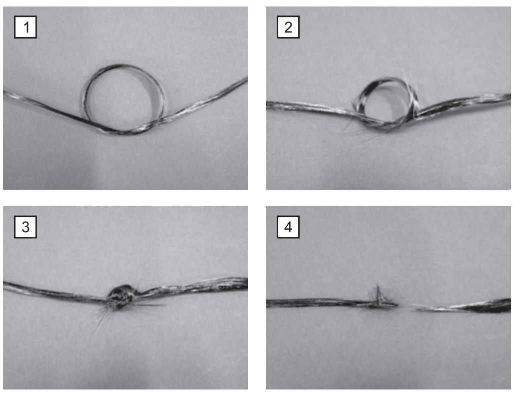

Figure 1. Knot test with pitch-based carbon fibre

Hence, a modification has to be found that avoids filament breakage during textile machinery processes. The characterisation for this modification can be done by two different standardisation tests from the German Institute for Standardisation, the Deutsches Institut für Normung (DIN). The first test is the knot- (DIN 53842) the second one is the loop tensile tests (DIN 53843).

2.1 Knot test

The knot test is used to determine the knot tensile strength $F_{H,K}$ at standard climate. The number of individual knot test is at least 20. The free length of the specimen is 500 mm. The strain rate is the deformation speed with which the clamps of the tensile testing machine remove from each other. In Table 2 the values of the strain rate dependent on the maximum tensile elongation of a test without a knot are given.

Table 2. Knot test strain rate [9]

Elongation at break, $_H$ [%]	Strain rate [mm/min]
Up to 5	50
Above 5 to 40	250
Above 40	500

For the experiment an overhand Z or S knot is used. Which of the two knots will be applied depends in general on the rotation direction of the fibres or the twist of the tail feather. For Z-rotation an S-knot and for S-rotation a Z-knot is used (Figure 2). Other selections have to be specified in the report.

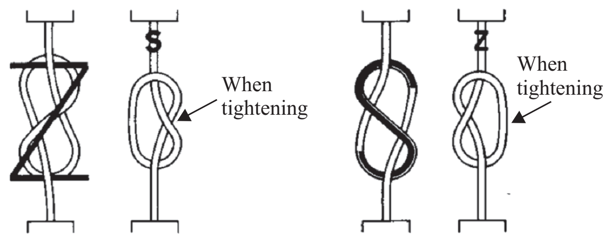

Figure 2 . Z-knot (left) and S-knot (right) [9]

The tests were performed using a STATIMAT 4U testing machine from Textechno, Mönchengladbach, Germany, using a measuring head with 10 N. Regarding Table 2 a strain rate of 500 mm/min is chosen.

2.2 Loop tensile test

The loop tensile strength is defined as $F_{H,S}$ and is measured during the test. The fineness-related loop strength $f_{H,S}$ is defined as the quotient from the loop tensile strength $F_{H,S}$ and twice the output fineness Tt of the broken fibre of the loop tensile test (Equation 2).

$$f_{H,S} = \frac{F_{H,S}}{2 \cdot Tt} \qquad (2)$$

The test sample is divided into two equal fibre sections. For the loop tensile test one of the two fibre sections is clamped as a loop in the upper clamp and the other one as a loop in the lower clamp of a tensile tester in such a way, that the two embrace each other in a predetermined way. The pulling clamp is moved at a predetermined speed. The tensile strength by the deformation of the two fibre sections is measured up to fracture of one or both sections. It is noted whether the fracture occurred at both sections or only one, and possibly at which.

The number of individual loop tensile tests is at least 20 and the clamping length is generally 20 mm. A defined preload force at the clamping of the two fibres is not required. The fibres have to be clamped so that after a short way of the pulling clamp (maximum 2 mm), a deformation of the fibre and thus a tensile force occurs. The two equal fibre sections are placed on a contrasting colour to the fibre surface crosswise (Figure 3 a). The two ends of the underlying fibres can be combined via the other fibre and clamped together with a pretension weight bracket (Figure 3 b). The other two ends of the fibre can be combined with a pair of tweezers and clamped in the tensile testing machine that the wrapping of the fibres is located in the middle of the distance between the clamps (Figure 3 c).

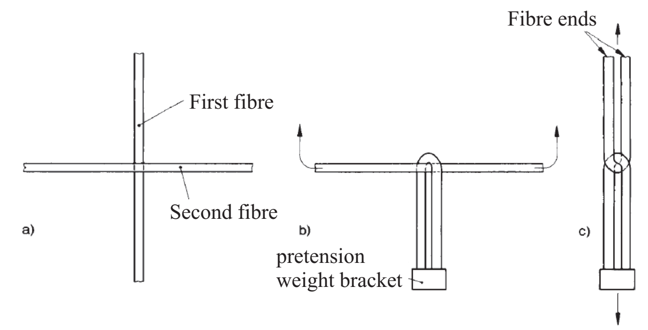

Figure 3. Preparation of fibre loops for tensile testing machine [10]

The fibre loops are fixed between the clamps of the tensile testing machine so that the fibre ends in each clamp are located directly beside each other (not stacked). The fibre ends of the lows in the upper and the lower clamp shall be placed in the middle of the clamps. When clamping it is important to note that the fibres are prepared with only a pair of tweezers at the fibre ends and not touched with the fingers.

3. RESULTS AND DISCUSSION

In the following chapter knot- and loop tensile test of selected pitch-based carbon fibres from Table 1 are discussed.

3.1 Knot test

The averaged knot test results of five different pitch-based carbon fibres related to the fibre fineness are given in Figure 4. The fibre "YSH-50A-10S" shows the highest strength with more than 0.12 cN/tex. The other fibres reach only values between 0.02 and 0.04 cN/tex. Overall these test results demonstrate the poor knot strength of all tested pitch-based carbon fibres.

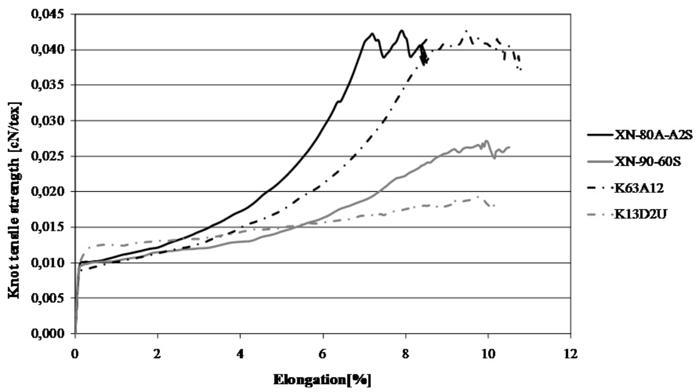

Figure 4. Knot test of different pitch-based carbon fibres

The fibre "XN-80A-A2S" was special modified at the ITA. Afterwards the knot test was performed and is given in Figure 5 in comparison to the virgin fibres.

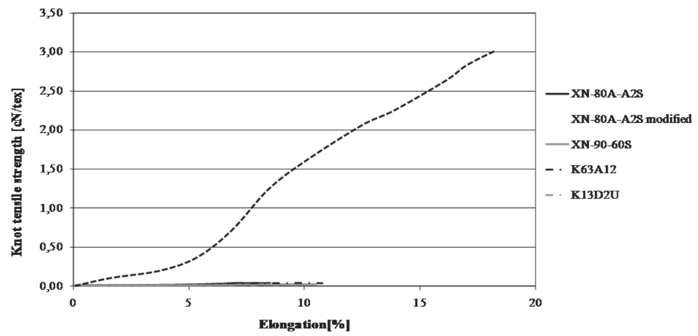

Figure 5. Comparison of knot test of different pitch-based carbon fibres before and after modification

The modified "XN-80A-A2S" achieves a strength increase of more than 70 times in the knot test while the elongation is increased too from 10 to almost 18 percent.

3.2 Loop tensile test

In comparison to the knot test the loop tensile test shows a smaller range of the fineness-related loop strength $f_{H,S}$ (Figure 6). The "XN-80A-A2S" reaches 5 cN/tex, followed by the "K63A12" with almost 4 cN/tex and the "YSH-50A-60S" with 3.3 cN/tex.

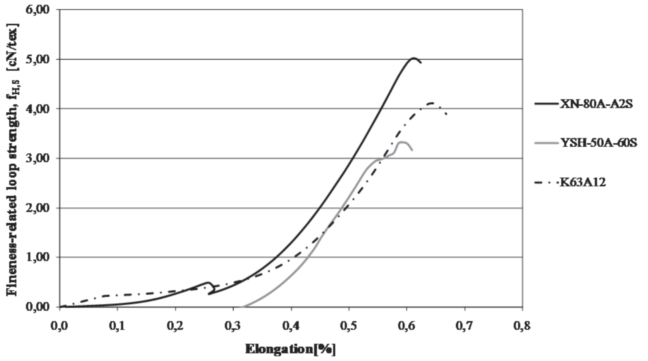

Figure 6. Loop tensile test of different pitch-based carbon fibres

The modification of the fibres before the loop tensile test results in a more than 16 times higher elongation combined with a strength increase of 20 % for the "XN-80-A2S" (Figure 7). The modification of the "YSH-50A-60S" achieves a slightly higher elongation with no change in the tensile strength of 3.2 cN/tex.

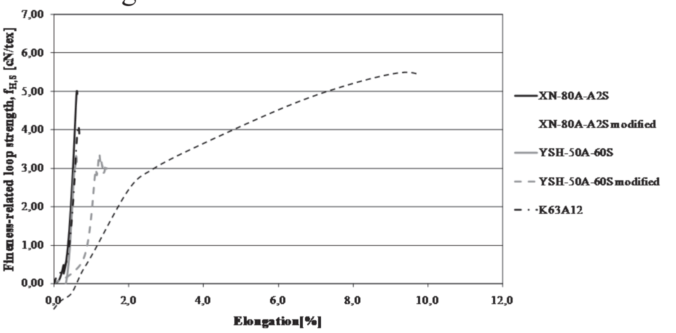

Figure7. Comparison of loop tensile test of different pitch-based carbon fibres before and after modification

Thus, the at ITA applied pitch-based carbon fibre modification is able to increase the knot strength and shows also for one of the investigated fibre materials a increase in the elongation for the loop tensile test. Further tests are carried out to investigate the influence of the modification of all fibres listed in Table 1.

4. CONCLUSION

The modification of very high-modulus pitch-based carbon fibres shows significant increases in the strength for knot tests of the pitch-based carbon fibre type "XN-80A-A2S" and significant increases in the elongation of the type "XN-80-A2S" as well as a slight increase in the strength. The type "YSH-50A-60S" shows only a slight increase in strength for the loop tensile tests. After the fibre modification FRP parts have been manufactured at ITA. The modified fibres were stitched into 2D-preforms in out-of-plane direction. Afterwards these preforms were impregnated using the Vacuum Assisted Resin Transfer Moulding (VARTM) process. The TC of these parts has been measured in in-plane- and out-of-plane direction and shows increases of 200 % and more compared to specimens without modified fibres [11].

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