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High strength and low weight hollow carbon fibres

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Abstract. Carbon fibres have strengths of 2.5 to 5 GPa in the fibre direction and an elastic modulus of 200 to 500 GPa. Carbon fibres have equal mechanical properties as steel but 20% of the weight. But the material is more expensive than steel. Therefore, they are only used in industry sectors where the benefits legitimate the high costs. The use of hollow rather than solid fibres allows an even lower weight of the components. At the same time, similar mechanical properties are achieved by the circular cross section. Carbon fibres are obtained from polyacrylonitrile fibers (PAN). These can be produced as hollow fibres. As a first step stabilization and carbonization of hollow PAN precursors is investigated to produce hollow carbon fibres.

1. Introduction

The efficient use of the remaining energy reserves is of great importance due to climate change. In vehicles, the use of low weight structures reduces fuel consumption to reduce CO₂ emissions. Carbon fibre reinforced plastic (CFRP) is suitable as a substitution material for metal constructions. The material is made of a composite of carbon fibres and a matrix. The fibres have a tensile strength of 2.5 – 5 GPa and an elastic modulus of 200 – 500 GPa [2]. Carbon fibres have equal mechanical properties as steel but 20% of the weight [4]. The material price of conventional carbon fibres is about 20 €/kg (the price of steel is about 0.5 €/kg) [5,6]. Therefore, they are only used in industry sectors where the benefits legitimate the high costs. The use of hollow fibres instead of solid fibres lead to an even lower weight of the components. Similar mechanical properties are achieved by the circular cross section. Carbon fibres are obtained from polyacrylonitrile fibres (PAN). These can already be produced as hollow fibres. The aim is to produce hollow carbon fibres from hollow PAN precursors.

[1] and [3] describe the production of fibres with a non-solid cross section. In [1] the PAN precursor is spun out with c-shaped cross section. It is stabilized at 200 - 300 °C and carbonized at 1000 - 2000 °C. It can be seen that the chemical reaction run faster than at conventional solid fibres [1]. In [3] the PAN precursor is spun out with several hollow cores to reduce the density of carbon fibres. It is managed with the island-in-a-sea method with a poly(methyl methacrylate) solution as island component. The fibres have a honey comp shaped cross section. It turns out that an amount of seven islands leads to a stable structure during stabilization and carbonization. The precursors are stabilized and carbonized with different temperature profiles. For a stable structure the stabilization was carried out in two stages. First the precursors were heated from room temperature up to 260 °C with a rate of 5 °C/min and held isothermally for 60 min. In the second stage the fibres were heated to 305 °C with a rate of 5 °C/min and held isothermally for 20 min. The carbonization is conducted



10 min at 1257 °C under argon atmosphere. The density of the produced fibres is at 1,2 g/cm³. The properties are compared with a conventional fibre (T300 with a density of 1,79 g/cm³) in table 1 [3].

Table 1. Properties of different trials of the honey comp fibres (1 - 4) and carbon fibre T300 [3]

trial	diameter [μm]	tensile strength [GPa]	density [GN*kg/mm]	related strength	E-modulus [GPa]	elongation at break [%]
1	8,3 ± 0,3	2,3 ± 0,8	1,92 ± 0,7		267 ± 96	0,9 ± 0,3
2	9,6 ± 0,3	2,4 ± 0,5	2,00 ± 0,4		342 ± 28	0,7 ± 0,1
3	10,1 ± 0,3	2,1 ± 0,5	1,75 ± 0,4		322 ± 25	0,6 ± 0,1
4	9,3 ± 0,3	2,4 ± 0,6	2,00 ± 0,5		368 ± 58	0,7
T300	7	3,5	1,96		230	1,5

It can be seen that the tensile strength of all fibres in relation to their density are in the same amount. The E-moduluses of the honey comb fibres are even higher.

Due to the previous works, some theses are formulated. In [3] the tensile strengths are compared. The bending stiffness is not tested. It can be assumed that there is a difference between hollow and solid fibres due to their different geometric moments of inertia. It is expected that hollow fibres have a higher bending stiffness in relation to their weight. In [1] was found that stabilization and carbonization of hollow fibres run faster. It can be assumed that the smaller cross section area is a reason. Also, hollow fibers have an additional surface inside where chemical reactions with oxygen can run. In [1] and [3] the non-solid precursors stay stable during stabilization and carbonization. It can be assumed that the production of hollow carbon fibres is possible.

2. Theoretical

The examination of mechanical properties is done theoretically. Bending stiffness and weight of hollow and solid fibers are compared. The bending stiffness is proportional to geometric moment of inertia. The weight is proportional to the cross-sectional area. According to the fibre diameters two functions are set up. The first function sets the diameters of hollow and solid fibres in relation while their weight is equal. the second function sets the diameters of hollow and solid fibres in relation while their bending stiffness is equal. They are shown in figure 1. The hollow fibres of the same weight as solid fibers have a higher bending stiffness. Also, it is possible to produce a hollow fiber with a higher bending stiffness and lower weight than a solid fibre.

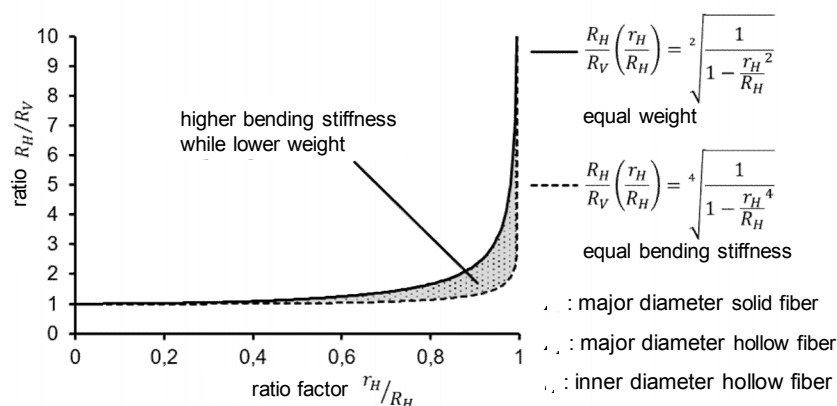


Figure 1. Bending stiffness and weight according to fibre diameters

3. Experimental

Two different PAN fibres are examined. The first fibre is purchased from MICRODYN-NADIR GmbH, Wiesbaden, Germany. It is used as a hollow precursor. It is hereinafter referred to as industrial fibre. The other hollow PAN precursor is spun out at the ITA and is referred to as ITA-fibre. To consider the surface and cross section of the precursors a Scanning Electron Microscope Leo (Zeiss) 1450 VP is used.

The fibres are stabilized discontinuously. Therefore a LM 312 laboratory muffle of LINN High Therm GmbH is used. Two trials are carried out for stabilization. The first test is made to analyse the behaviour of the fibre structure under the influence of different temperatures. The fibres are stabilized with three different temperature profiles. The first temperature profile is defined by the ITA and is based on the experience with the production of carbon fibres. The starting temperature is 160 °C. and the final temperature is 260 °C. A constant heating rate of 1 °C./min is used. It is referred to as 260 °C-profile. As described carbon fibres with honey comp cross section are produced in [KNG13]. The described temperature profiles with a final temperature of 305 °C is used for comparison and referred to as 305 °C-profile. For the third temperature profile, the 260 °C-profile is used with an adjusted trial time. A correction factor c is determined by the following equation:

$$c = 1 - \frac{d^2}{D^2}$$

where d is the inner diameter of the fibre and D the major diameter of the hollow fibres. The adjusted trial time is determined by following equation:

$$t_{i,corr} = t \cdot c_i$$

where t is the trial time of the 260 °C-profile and is set to 100 min. With the measured diameters for the industrial fibre a factor c of 0.67 is determined and an adjusted trial time of 67 min. For the ITA-fibre a factor c of 0.53 is determined and an adjusted trial time of 53 min.

Furthermore, it is stated that hollow fibres have an additional surface inside, on which reactions can take place. Oxygen in the core of the fibres is necessary so that dehydration and oxidation can take place. To investigate this effect, a second trial is carried out. It is investigated whether the reactions in the interior are weaker when the oxygen is cut off. For this purpose, the fibre ends are closed airtight at one sample each. A sample with open fibre ends serves as a comparison. The fibres are stabilized with the 260 °C profile.

For discontinuously carbonization a tube furnace from type FRH-70/500/1600 is used. Fibre samples stabilized with the 260 °C-profile are carbonized at 1200 °C under nitrogen atmosphere. To microscopy all produced fibre samples a stereo microscope from type Leica M205C is used.

4. Results and discussion

At first the cross-sections of the precursors are watched under the scanning electron microscope (SEM). In figure 2 both precursors have a circular cross section with a hollow core in the center.

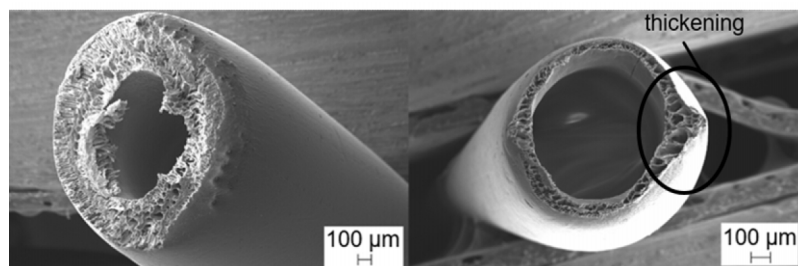


Figure 2. Cross sections of precursors under SEM; industrial fibre (left) and ITA-fibre (right)

The ITA-fibre has a thicker wall on one side. Both precursors do not have a homogeny cross section area but show many hollow spaces between inner and outer surface. The industrial fibre has an average inner diameter of 1,2 mm and major diameter of 2,08 mm. The ITA-fibre has an average inner diameter of 0,78 mm and major diameter of 1,18 mm.

In figure 3 the industrial fibre is pictured after 50% and 100% trial time of stabilization. In all cases the hollow structure stays stable. During stabilization, there is formed a core-mantle structure in the cross-section area. The core area is surrounded by two mantle areas. These are in contact with oxygen during the process. This leads to the assumption that the reactions with oxygen are the reason for building this structure. Also it can be seen that the fibres changes the color from white to black with higher temperature. In case of the 305 °C-profile, 260 °C are reached after 50% of time. The color of the fiber is quite different to the other samples at this time. This suggests that the stabilization process may be completed after 50% and the final temperature is set too high.

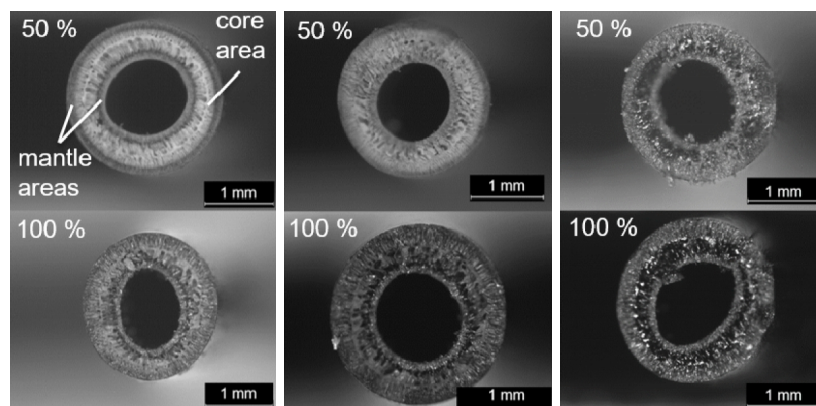


Figure 3. Industrial fibre after 50% and 100% trial time of stabilization; 260 °C-profile (left), adjusted 260 °C-profile (center), 305 °C-profile (right)

In figure 4 the ITA-fibre is pictured after 50% and 100% trial time of stabilization. As in the case of the industrial fibre no change of the hollow structure can be seen. The core-mantle structure is formed too. The effect of color changing is shown too. Unlike the industrial fibre the ITA-fibre shows a thickening in the wall at one side. It can be seen at the precursor too which means, it does not appear during the stabilization.

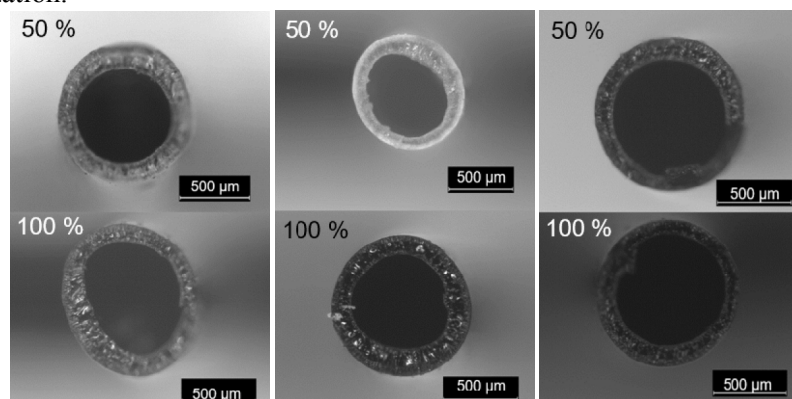


Figure 4. ITA-fibre after 50% and 100% trial time of stabilization; 260 °C-profile (left), adjusted 260 °C-profile (center), 305 °C-profile (right)

In the second trial samples with airtight closed and open fibre ends are compared. After stabilization, there is no difference between both. Two explanations are possible. First the oxygen

locked inside after closing the fibre ends could suffice for the reactions with oxygen. The other explanation is that oxygen still streams through the fibre walls by diffusion. The hollow spaces seen in figure 2 could allow oxygen to diffuse fast to the inside.

In figure 5 cross-sections of the industrial fibre after carbonization are pictured.

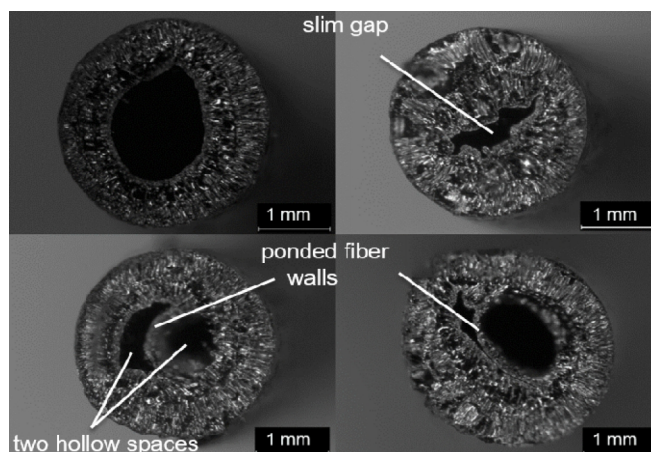


Figure 5. Cross-sections of the industrial fibre after carbonization at 1200 °C

In some cases, there are changes in the structure after carbonization. In some cases, the circular core transformed into a slim gap. In some cases, the inner fibre walls cut off and created two hollow spaces in the center. One reason could be the high mass loss during carbonization which can be up to 40%. This leads to inner tensions and to a deformation of the geometry. Another explanation are gases which are ejected during carbonization. It is conceivable that these gases pond in some hollow spaces, create high pressure and blow up the inner fibre walls.

In figure 5 the cross-section of the ITA-fibre after carbonization is pictured. Unlike the industrial fibre there are no transformations after the carbonization. One reason for this difference could be the lower wall thickness of the ITA-fiber. Thus, the ejected gases can diffuse faster to the outside and do not pond.

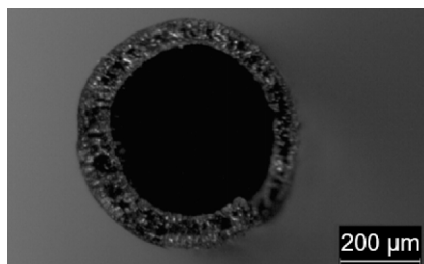


Figure 6. Cross-section of the ITA-fibre after carbonization at 1200 °C

5. Conclusion

The results show that the hollow fiber structure is largely retained during the stabilization reactions. Small irregularities, which can be seen after stabilization, also occur in the precursor. This means that they do not appear during stabilization. The hollow fibers form a core-mantle structure with three areas. There is no difference between fibers with open and closed fiber ends. In the case of carbonization, some samples show changes in the geometry.

Stabilization and carbonization are carried out discontinuously in the laboratory scale. In continuing projects the continuous process should be considered. For this, it must be examined whether air diffuses through the fiber wall or not. One possibility is to separate the three regions of the core-mantle structure and compare their chemical composition. Interesting is also whether nitrogen

diffuses through the stabilized fibers. The carbonization should take place under an inert atmosphere to prevent reactions with oxygen. Incorporated oxygen in the fiber interior can lead to an undesirable effect in carbonization. Furthermore, a check of the mechanical properties of the hollow carbon fibers must follow to validate the theoretical statements.

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