



Carbonization of special viscose fibers

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The focus of the work was to clarify the influence of the cross section shape of the viscose fibers and additives on the carbonization process and the resulting carbon fiber properties. Overall, the shape of cross section of carbon fibers produced was determined by the initial fiber morphology of the precursor viscose fibers. The carbon yield was independent of the geometric structure of the viscose fibers and a certain degree of shrinkage was observed for all types of fibers due to the loss of water, hydrocarbons and other volatile products. Several additives, such as carbon black, a nitrogen containing carbohydrate and lignin, were homogeneously incorporated into the precursor fiber and resulted in slight increase of the carbon yield. Carbon black particles became distinctive and visible on the surface of the carbon fibers. In particular, the thermal and chemical course of the carbonization was altered by the nitrogen containing additive, while the structure and properties of the carbonized fibers remained basically unchanged.

Introduction

Carbon fibers are accessible from several raw materials [1–3]. The industrial most important precursor fiber is Polyacrylonitril (PAN), which delivers carbon fibers with extremely high modulus and tenacity. The major application of these fibers are fiber-reinforced composites for lightweight construction, e.g. in the fields of automotive, aviation, wind power plants and sports goods.

However, the first commercially available carbon fibers were based on cellulosic precursor fibers and were used in carbon filament lamps. Nowadays regenerated cellulosic fibers like viscose are still used as precursors for carbon fibers. A major application are nonwovens for high-temperature isolation (Figs. 1 and 2), e.g. in furnaces for the production of wafers for solar cells.

The carbonization of the cellulosic viscose fibers is a pyrolytic process in which theoretically just water is demerged and pure carbon is formed (Fig. 3). With increasing temperature, the carbon structure develops toward graphite structure.

The calculated maximum carbon yield is 44%, in this case just water would be demerged during carbonization and graphitization. In reality the carbon yield is much lower since carbon containing molecules are emerged as volatile side products: carbon monoxide

and dioxide, hydrocarbons and oxygen containing organic compounds [1].

Results and discussion

Variation of shape of fiber cross section

To study the influence of the fiber cross section on the carbonization process different viscose fibers with round trilobal and flat cross section were tested (Fig. 4).

The scanning electron microscopy images clearly show that the fiber diameter of carbonized fibers was much smaller than the diameter of the initial precursor viscose fibers (Fig. 4). The loss of carbon containing molecules and water revealed a certain shrinkage. It is remarkable that the characteristic morphology and geometric structure of the fibers was retained. All carbonized fibers showed specific cross sections as well as the typical structured surface structure, which was considerably pronounced in the fiber with round cross section.

Preparative determination of the carbon yield was carried out with carded needle punched nonwovens. These fabrics were carbonized in a high temperature furnace under an inert gas atmosphere. The carbon yield was largely independent of the shape of the

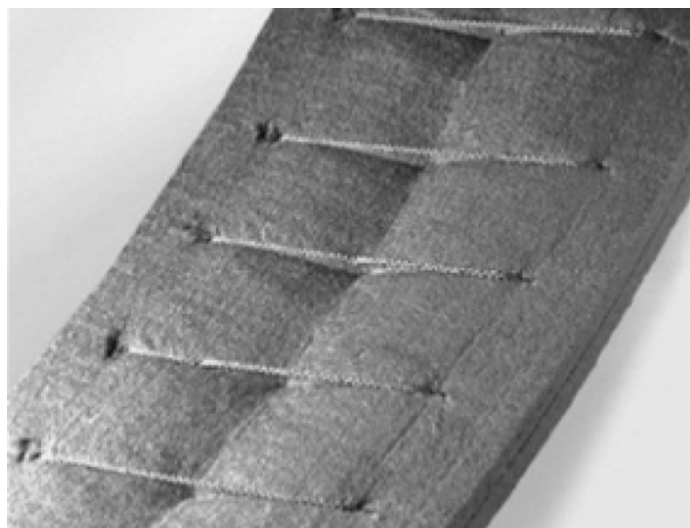


FIGURE 1

(Courtesy of SGL Carbon): Nonwoven of carbon fibers for thermal isolation.



FIGURE 2

(Courtesy of SGL Carbon): High temperature oven.

fiber cross section (Table 1). Carbonization up to 1800°C of all fabrics, consisting of viscose fibers with either round, trilobal or flat cross sections, resulted in carbon yields in the range of 14–15%. The data in Table 1 show that the carbonization was already almost complete at 900°C. However, the data indicate a slightly higher carbon yield for round shaped fibers compared to trilobal and flat fibers.

Analytical investigation of the carbonization with thermogravimetric analysis (TGA) showed for all three fiber types similar curves of remaining mass as a function of rising temperature (Fig. 5). Again a slightly higher carbon yield for round shaped fibers compared to trilobal and flat fibers was observed. The carbon yields of 21–24% in the TGA analysis were considerably higher than the preparative yields of 14–15% as aforementioned due to different set ups and conditions during carbonization. The major part of the mass loss appeared in the temperature range between 250 and 350°C. The final level of carbon yield is more or less reached at 900°C. Furthermore, DTA and DSC experiments were performed in order to investigate the thermal stability and decomposition processes. As shown in Fig. 6, the decomposition of cellulose occurred at the same temperature independent of the cross section indicated by the endothermic peaks at 360°C.

During carbonization and graphitization of cellulose different structures and chemical bonding of the carbon atoms are passed.

In the cellulose the carbon atoms form four covalent bonds with three-dimensional tetrahedral direction (sp^3 -hybridized carbon, Fig. 7). In graphite the carbon atoms form just three main bonds with quasi-two-dimensional trigonal-planar direction (sp^2 -hybridized carbon). A chemical structure is formed with carbon layers which are less strong bonded to each other than the carbon atoms within one layer. Depending on a tetrahedral or planar configuration of the chemical bonding, different vibrational behavior of the carbon atoms in the lattice of a carbonized material occurs. Reversely it is possible to get information about the bonding structure of the carbon atoms from their vibrational behavior, which can be investigated with Raman spectroscopy.

According to the Raman spectra in Fig. 8 the bonding structure of the carbon atoms and the supramolecular structures within the different fibers are similar. The band located at 1600 cm^{-1} is the G mode of graphite, which is characteristic for the motion of sp^2 -hybrids of carbon in the graphite planes. As for all less ordered carbon materials, a defect induced D-band was found at 1350 cm^{-1} . Thus, the carbonization up to 900°C did not reveal fibers with pure graphite structure. Increase of the carbonization temperature up to 1800°C leads to stronger graphitization, observable in the more distinct separation of the D-band and the G-band

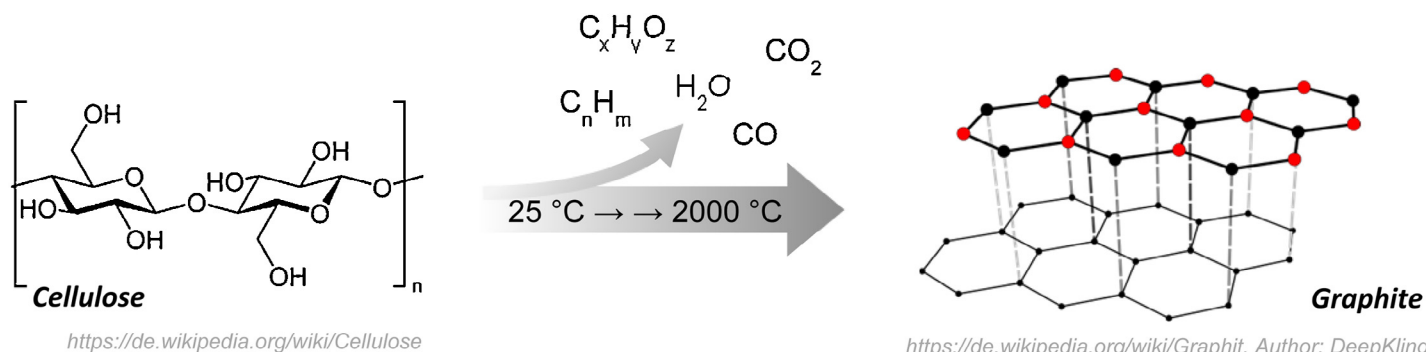


FIGURE 3

Carbonization of cellulose (viscose).

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